The Journal of the Society of Dyers and Colourists

Volume 74



Number 9

CONTENTS

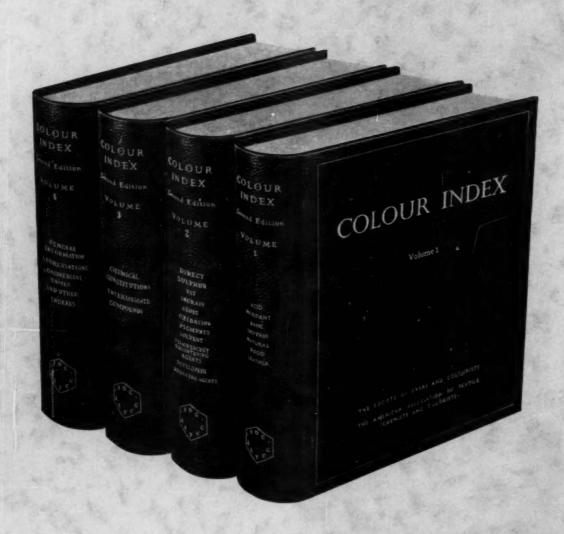
EXAMINATION FOR THE ASSOCIATESHIP (A.S.D.C.) 1958	
Examination Papers	621
REPORT OF THE EXAMINATIONS BOARD	625
PROCEEDINGS OF THE SOCIETY	
THE DYEING OF ACRILAN ACRYLIC FIBRE (H. R. Hadfield and W. M. Sokol)	629
RECENT DEVELOPMENTS IN THE APPLICATION OF AZOIC DYES (M. Hückel)	640
COMMUNICATION	
Adsorption at Organic Surfaces III—Some Observations on the Constitution of Chitin and on its Adsorption of Inorganic and Organic Acids from Aqueous Solution (C. H. Giles, A. S. A. Hassan, (Miss) Margaret Laidlaw, and	
R. V. R. Subramanian)	647
CORRESPONDENCE	655
NOTES	656
NEW BOOKS AND PUBLICATIONS	657
ABSTRACTS	658

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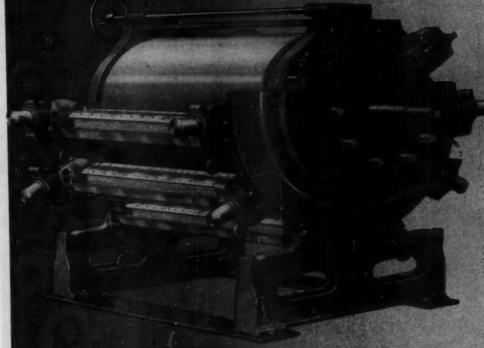
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LONDON SECTION

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The Journal of the Society of Dyers and Colourists

• (Subscription rates for non-members £5 5s 0d per annum, post free) (Abstracts section only printed on one side of paper—£2 0s 0d per annum)

NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring general information regarding the Official Notices, List of Officers of the Society, etc, should consult pages 1-7 of the January 1958 and pages 509-516 of the July 1958 issues of the Journal, or write to The General Secretary, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (Telephone Bradford 25138). Editorial Communications should be addressed to The Editor, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the Journal —

LECTURES

Recent Developments in Dyeing for the Carpet Trade

W. Beal

The Determination of Damage to Wool Fibres

7. C. Brown

Colour in Writing and Copying

J. P. Gill

The Continuous Dyeing of Wool

D. R. Lemin

Water-repellent Finishes-Modern Use of Silicones

G. W. Madaras

Flameproofing of Textile Fabrics with particular reference to the Function of Antimony Compounds

N. J.

N. J. Read and E. G. Heighway-Bury

COMMUNICATIONS

The Dyeing of Secondary Cellulose Acetate with Disperse Dyes VII— A Comparison with their Solubility in Organic Solvents

C. L. Bird

- Adsorption at Organic Surfaces-
 - IV-Adsorption of Sulphonated Azo Dyes on Chitin from Aqueous Solution

C. H. Giles, A. S. A. Hassan, and R. V. R. Subramanian

V- A Comparison of the Adsorption of Solutes by Cellulose and Chitin

C. H. Giles and A. S. A. Hassan

Unlevel Dyeing in Wool Velour Cloth

F. J. Parker

EXPLANATORY PAPER ON MODERN THEORY

Surface Activity, Solution, and Adsorption

C. H. Giles

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Friday evening 18 September 1959

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Already heavy advance reservations have been received for tickets and all interested are invited to communicate with —

THE GENERAL SECRETARY
THE SOCIETY OF DYERS AND COLOURISTS

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FORTHCOMING MEETINGS OF THE SOCIETY

Tuesday, 23rd September 1958

Scottish Section. Ladies Evening. The Good House-keeping Institute and its Work. Miss A. M. Kaye. St. Enoch Hotel, Glasgow. 7.15 p.m.

Wednesday, 24th September 1958
LONDON SECTION. Surface Activity: The General Concept
and Some Consequences. Dr. J. L. Moilliet (Imperial
Chemical Industries Ltd., Dyestuffs Division). Joint
Meeting with the London Section of the Oil & Colour Chemists' Association.) Refreshments will be served at 6.15-6.45 p.m. in the Library. Manson House, 26 Portland Place, London W.1. 7 p.m.

Wednesday, 1st October 1958

MIDLANDS SECTION. Dyeing Unions containing Acrylic Fibres. B. Kramrisch, Esq., F.R.I.C., F.T.I., F.S.D.C. College of Technology, Leicester. 7 p.m.

Friday, 3rd October 1958

LONDON SECTION. Plastics and their Colourings. C. Musgrave, Esq., A.M.C.T. (Geigy Co. Ltd.). Royal Society, Burlington House, London W.1. 6 p.m.

Wednesday, 8th October 1958
SCOTTISH JUNIOR BRANCH. Colour Matching. E. Bellhouse,
Esq., B.Sc. (The Geigy Co. Ltd.). Technical College,
George Street, Paisley. 7.30 p.m.

Friday, 10th October 1958

WEST RIDING SECTION. Supper and Smoker. Victoria Hotel, Bridge Street, Bradford.

Saturday, 11th October 1958

MIDLANDS SECTION. Ladies Evening-Dinner Dance. Grand Hotel, Leicester.

Tuesday, 14th October 1958
Bradford Junior Branch. Colour Matching. E. Bellhouse,

Bradford Junior Branch. Colour Matching. E. Bellhouse, Esq., B.Sc. (The Geigy Co. Ltd.). Institute of Technology, Bradford. 7.15 p.m.

Leeds Junior Branch. Film Show. (1) Nagara (Gy.). (2) The Discovery of a New Pigment (I.C.I.). (3) How the World Looks to a Colour Defective. Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, Leeds. 3.15 p.m.

Northern Ireland Section. The Dyeing of Terylene/Cellulosic Fibre Blends. J. G. Graham, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Grosvenor Rooms, Belfast. 7.30 p.m.

Belfast. 7.30 p.m.

p.m.

Thursday, 16th October 1958

MANCHESTER JUNIOR BRANCH. Dyeing the new Fibres.
B. Kramrisch, Esq., F.R.I.C., F.T.I., F.S.D.C. College of Science and Technology, Manchester. 4.30 p.m.

Friday, 17th October 1958

MANCHESTER SECTION. Some Recent Advances in Textile
Printing. R. J. Hannay, Esq., B.Sc., F.R.I.C., F.T.I.,
F.S.D.C. (Brotherton & Co. Ltd., Leeds). The Textile
Institute, 10 Blackfriars Street, Manchester. 7 p.m.

Tuesday, 21st October 1958

HUDDERSFIELD SECTION. Methods of Assessing the Dyeing Properties of Wool Dyes. B. Kramrisch, Esq., F.R.I.C., F.T.I., F.S.D.C. (Ciba Clayton Ltd.). (Silvios Cafe, Huddersfield. 7.30 p.m.

Scottish Section. A Survey of the Various Methods of Continuous Dyeing. Dr. H. Waibel (Badische Anilin & Soda Fabrik A.G.). St. Enoch Hotel, Glasgow. 7.15

Wednesday, 22nd October 1958
BRADFORD JUNIOR BRANCH. The Properties of Fibres in Relation to Chemical Structure. Dr. A. Sharples (British Rayon Research Association). Institute of Technology, Bradford. 7.15 p.m.

Thursday, 23rd October 1958
West RIDING Section. The Solubility of Wool Dyes—Its
Assessment and Practical Significance. W. Beal, Esq.,
B.Sc. (The Geigy Co. Ltd.). Victoria Hotel, Bridge
Street, Bradford. 7.30 p.m.

Friday, 24th October 1958

London Section. The Sixth London Lecture. Colour in Art. A. Lucas, Esq. (Chief restorer to the National Gallery). The Waldorf Hotel, London W.C.2. 7 p.m. NORTHERN IRELAND SECTION. Dinner Dance. Woodbourne House Hotel.

Tuesday, 28th October 1958

Leeds Junior Branch. Developments in Modern Dry Cleaning Techniques. E. J. Davies, Esq., M.Sc. Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, Leeds. 3.15 p.m.

Wednesday, 29th October 1958
Bradford Junior Branch. Visit to Stevensons (Dyers) Ltd., Ambergate.

MIDLANDS SECTION. The Setting, Dyeing and Finishing of Banlon Knitted Goods. (Lecturer to be announced later.) King's Head Hotel, Loughborough. 7 p.m.

Thursday, 6th November 1958

Bradford Junior Branch. Dyeing Unions Containing Acrylic Fibres. B. Kramrisch, Esq., F.R.I.C., F.T.I., F.S.D.C. (Ciba Clayton Ltd.). Institute of Technology, Bradford. 7.15 p.m.

Friday, 7th November 1958

Friday, 7th November 1958

LONDON SECTION. Investiture of the Chairman's Badge by the President. Some Aspects of the Dyeing and Finishing of Fabrics Knitted from Bulky and Stretched Yarns. S. M. Jacckel, Esq., B.Sc., A.R.C.S., A.R.I.C., F.C.S., and D. L. Munden, Esq., B.Sc. (The Hosiery & Allied Trades Research Assoc.). The Royal Society, Burlington House, London W.1. 6 p.m.

WEST RIDING SECTION. ONE DAY CONFERENCE. Dyeing Unions containing Acrylic Fibres. B. Kramrisch, Esq., F.R.I.C., F.T.I., F.S.D.C. (Ciba Clayton Ltd.). Dyeing of Polyester Fibres and Blends. H. W. Partridge, Esq., M.Sc., A.T.I. (Stevensons (Dyers) Ltd.). Finishing of Blended Fabrics containing Wool and Acrylic or Polyester Fibres. C. S. Whewell, Esq., B.Sc., Ph.D., F.R.I.C., F.T.I., F.S.D.C. (Leeds University). The Uses of Acrylic Fibre Blends with reference to Colour Fastness. H. D. Edwards, Esq., B.Sc., Ph.D., A.R.I.C. (Courtaulds Ltd.). (Courtaulds Ltd.).

Institute of Technology, Bradford. 2 p.m.

Tuesday, 11th November 1958
LEEDS JUNIOR BRANCH. Theoretical Aspects of Solution
Chromatography. Dr. A. Levi (Imperial Chemical
Industries Ltd.). Lecture Theatre, Department of
Colour Chemistry and Dyeing, The University, Leeds. 3.15 p.m.

NORTHERN IRELAND SECTION. General Principles in Pig-ment Padding with Vat Dyes. Dr. R. Kern. (Sandoz, Basle). Grosvenor Rooms, Belfast. 7.30 p.m.

Thursday, 13th November 1958
WEST RIDING SECTION. The Appearance of Textile Faults in Dry Cleaning. (A member of the Dyers and Cleaners Research Organisation.) Griffin Hotel, Boar Lane, Leeds. 7.30 p.m.

Tuesday, 18th November 1958

HUDDERSFIELD SECTION. The Dry Cleaning of Textiles. E.
J. Davies, Esq., M.Sc. (Dyers & Cleaners Research
Organisation). Silvios Cafe, Huddersfield. 7.30 p.m. MIDLANDS SECTION. The use of Anti-static Agents in Textile Processing. A. E. Henshall, Esq., B.Sc. (Joint meeting with the Nottingham Textile Society.) Gas Board Theatre, Nottingham. 7 p.m.

Scottish Section. New Levelling Agents in Acid and Direct Dyebaths. G. H. Lister, Esq., B.Sc., Ph.D. (Sandoz Products Ltd.). St. Enoch Hotel, Glasgow. 7.15 p.m.

Wednesday, 19th November 1958

Scottish Junior Branch. Some Textile Dyeing and Drying Machines. K. S. Laurie, Esq., A.M.I.Mech.E., A.M.I.E.E. (John Dalglish & Sons Ltd.). (This lecture will be illustrated by means of a film show.) Technical College, George Street, Paisley. 7.30 p.m.

Thursday, 20th November 1958

London Section. Developments in Modern Dry Cleaning Techniques. E. J. Davies, Esq., M.Sc. (Dyers & Cleaners Research Organisation). (Joint meeting with the London Centre of the Guild of Dyers & Cleaners.) Institute of Journalists, 2-4 Tudor Street, London E.C.4. 7.15 p.m.

MANCHETER JUNIOR BRANCH. Textile Microsopy. G.

MANCHESTER JUNIOR BRANCH. Textile Microscopy. G. Cartridge, Esq., College of Science and Technology, Manchester. 4.30 p.m.

continued on page XXXIX



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THE JOURNAL

OF THE

Society of Dyers and Colourists

Volume 74 Number 9

SEPTEMBER 1958

Issued Monthly

Examination for the Associateship (A.S.D.C.) 1958

The fifth examination for the Associateship of the Society of Dyers and Colourists was held in May 1958, and below appear the question papers and the report of the Examinations Board. It is hoped that publication of these papers and the report will be helpful both to prospective candidates and to teachers, and that it will serve to inform members of the Society and others of the standard of knowledge expected from candidates for the Associateship.

Examination Papers

Papers A and B are taken by all candidates; but Papers C, D, and E are taken by a candidate only in his chosen branch of tinctorial technology. Further, in Paper E a candidate has a choice of subject—analytical and testing techniques or the organisation of production and economics

Paper A

General Chemistry and Properties of Dyes, Pigments, Auxiliary Products General Chemistry and Properties of Organic High Polymers

10 a.m.-1 p.m. on Thursday, 29th May 1958

(SIX questions only to be attempted, three from each section of the paper)

Important—Candidates must keep their answer papers to Sections I and II separate, and must mark clearly the Section to which each set of answers refers. They must not include parts of answers relating to Sections I and II on the same sheet of paper.

SECTION I

- 1. Compare the characteristic properties of acid dyes of the triarylmethane and the anthraquinone groups. Outline the preparation of one acid dye of each group.
- 2. Outline the chemical structure and discuss the mode of action of auxiliary products used (a) for aftertreating cellulose dyed with direct dyes to improve wet fastness, and (b) as scouring assistants.
- 3. Describe the preparation of phthalic anhydride and its conversion into anthraquinone compounds used in the dyemaking industry.
- 4. Write a short account of the indigoid dyes, including a modern synthesis of indigo.
- 5. Write an essay on the development of disperse dyes for secondary cellulose acetate with

reference to the influence of chemical structure on the fastness to—

- (a) Light
- (b) Wet treatments
- (c) Burnt-gas fumes
- (d) Sublimation.
- 6. Discuss the chemistry of the diazotisation process.
- 7. State the methods used for introducing the hydroxyl group into the aromatic nucleus, and give typical examples in the benzene, naphthalene, and anthraquinone series.
- 8. The ease of replacement of a chlorine atom in a benzene nucleus is influenced by the presence of other groups in the nucleus. Comment on this with reference to the formation of nitrophenols and nitroamines of the benzene series.

SECTION II

- 9. What methods may be used to cross-link linear polymers? By means of examples indicate what effects cross-linking may have on the physical properties of a polymer.
- 10. Distinguish between atactic, isotactic, and syndiotactic polymers, and state how isotactic polymers may be prepared.
- 11. Write concise explanatory notes on each of the following—
 - (a) Cationic polymerisation
 - (b) Azeotropie copolymer
 - (c) Block copolymer
 - (d) Emulsion polymerisation.

- 12. Write an account of **either** the structure of urea-formaldehyde resins **or** chain folding in proteins.
- 13. How may measurements of osmotic pressures of dilute solutions of a high polymer be used to obtain the molecular weight of the polymer? Describe briefly one method by which the osmotic pressure of a dilute solution of a high polymer may be measured.
- 14. Write a short essay on **one** of the following topics—
 - (a) Polyamides
 - (b) Vinyl polymers
 - (c) Polymer degradation.

Paper B

Theories of Colour Relation of Colour to Constitution Colour Assessment and Colour Fastness

2-5 p.m. on Thursday, 29th May 1958

(FIVE questions only to be attempted)

- 1. Describe the principles of a spectrophotometer. Explain what is meant by band width and stray light, and indicate how these affect the accuracy of an instrument when it is used for measuring optical density. How would you verify that the optical density scale of a particular instrument is correct?
- 2. Two dyes, A and B, in aqueous solution have the absorption characteristics shown in the table below, and the absorption is not affected by temperature. The absorption spectrum of a mixture of A and B does vary with temperature. Optical densities at 20°c. and 80°c. are given in the table. What are the colours of A and B; what are the concentrations present in the mixture; and what other conclusions may be drawn from the data?

Wavelength, mµ.
420 440 460 500 540 580 600 620 660

A (1 g./litre) 0-87 0-83 0-67 0-19 0-04 - - -

B (1 g./litre) 0·22 0·29 0·32 0·32 0·45 0·64 0·67 0·60 0·17

Mixture at 20°C. 1·31 1·27 1·10 0·84 0·61 0·62 0·62 0·58 0·17

Mixture at 80°c. 1-53 1-54 1-33 0-60 0-51 0-64 0-67 0-60 0-17

3. You are asked to illustrate, by means of coloured patterns in a systematic arrangement, the range of colours which can be produced by using either three selected dyes applied to textile fabric or three selected pigments applied as water paints. How would you select the three dyes or pigments to give the maximum range of mixed colours? Describe the system in which you would arrange the patterns and the practical methods which you would use to achieve a fairly uniform visual spacing of these patterns.

- 4. Many dyes, pigments, and colourless substances absorb infrared and ultraviolet radiation in addition to visual radiation. How does this arise in the absorbing molecule; how (briefly) is the absorption measured; and what practical applications are made of non-visual absorption in the colour-using trades?
- 5. Describe any published investigation into dyeing or pigmenting processes, or into the determination of fastness properties, which has depended largely on the use of spectrophotometric or colorimetric measurements. Explain the advantages of using such methods in the example chosen.
- 6. Discuss the difficulties of determining colorimetrically the amount of dye present in **either** a dyed fabric **or** an opaque pigmented plastic, as compared with estimation in solution. What method would you adopt in the example chosen and why?
- 7. Your company has been asked by a Government Department to specify the colour of a material in terms of physical quantities which can be measured on an instrument to avoid dispute. How would you set up such a specification, and how would you ensure that the specification was one that could be met by the works?
- 8. In using a colour-filter type of photoelectric colorimeter, it is found that the optical densities of solutions of a direct dye in water are not strictly proportional to the concentration of dye present. What are the possible causes of such anomalous behaviour, and what steps would you take to avoid it?
- 9. Discuss, in terms of modern electronic theory, the origin of colour in **one** of the following groups of dyes—
 - (a) Cyanine dyes
 - (b) Di- and tri-phenylmethane dyes
 - (c) Indigo and indigoid dyes.
- 10. An artist presents to you a colour circle made up of 24 coloured paper patterns, for which he makes the following claims—
 - (a) Colours diametrically opposite in the circle are complementary
 - (b) The hue of the afterimage of any colour is that of the colour diametrically opposite
 - (c) The visual spacing of the colours around the circle is equal; i.e. the visual colour difference between any two adjacent patterns is the same
 - (d) If water paints are prepared to match any two diametrically opposed colours, then these paints, when mixed in suitable proportions, will give a neutral grey.

Assuming that claim (a) is valid, which, if any, of the remaining claims are likely to be invalid and why? How would you confirm experimentally that claim (a) is in fact valid?

11. Who is the "Standard Observer" in the C.I.E. system of colorimetry and how is he defined?

What are the standard light sources and the standard viewing conditions in this system?

12. You are asked to investigate the rate of dyeing of wool-nylon unions with acid dyes, determining at all stages the distribution of dye between the two fibres. Describe the method which you would use, assuming that the union fabric may be replaced by equivalent proportions of separate wool and nylon fabrics for the purpose of the investigation.

BRANCH 1— TINCTORIAL TECHNOLOGY AS APPLIED TO TEXTILES

Paper C

Properties of the Materials and Finished Products and Fundamental Mechanisms of the Industrial Processes

10 a.m.-l p.m. on Friday, 30th May 1958 (SIX questions only to be attempted)

- 1. Discuss critically work published in the last few years on **either** (i) the effect of temperature and humidity on the fading of dyed textiles **or** (ii) the dyeing of secondary cellulose acetate with disperse dyes.
- 2. Either (i) outline the principles which underlie the application of vat dyes to cellulosic fibres with particular reference to dyeing from a reduced bath, padding with a reduced dye, and application of the dye as unreduced pigment, and comment critically on each kind of method; or (ii) discuss the significance of leuco-potential measurements in the printing of vat dyes in an alkali-formaldehyde-sulphoxylate style.
- 3. What is meant by the term carrier when applied to a dyeing assistant? What substances may be used as carriers for dyeing hydrophobic fibres and how do they work?
- 4. Discuss the dyeing behaviour of the following types of fibres with particular reference to the chemical constitution of the molecule and the fine structure of the fibre—
 - (a) Polyacrylonitrile
 - (b) Polyester
 - (c) Regenerated protein.
- 5. What chemical and physical changes are believed to take place when wool is—
 - (a) Exposed to light
 - (b) Chlorinated
 - (c) Carbonised?

Discuss the influence of these changes on the dyeing behaviour of the fibre.

- 6. How may the molecular structure of fibrous cellulose be modified by the action of oxidising agents commonly used for bleaching? How are these modifications reflected in—
 - (a) The dyeing behaviour of the fibre
 - (b) The properties of the dyeing?

- 7. What kinds of attractive forces are considered to bond dye molecules to fibre substances? Outline the characteristics of each kind of force and show how they may determine dyeing behaviour and fastness properties in a particular dye-fibre system.
- 8. How is the mechanism by which starch is degraded in treatments with amylase preparations related to the source of the active material and the conditions under which it is employed?
- 9. Discuss attempts that have been made to relate the flow properties of printing pastes with their behaviour during printing and with the quality of the print.
- 10. What types of product are formed by the action of reducing agents on typical dyes of the azo and of the anthraquinone series? How are the reactions employed in textile dyeing and printing?
- 11. Outline methods by which the pH value of solutions may be measured, and discuss the value of such measurements to the dyer.
- 12. Describe qualitatively the effect of temperature on the rate of dyeing of a substantive dye from a molecularly dispersed solution on an appropriate textile fibre. What is the significance of the activation energy of the process, and what steps would need to be taken in order that it might be evaluated for a given dyeing system?

Paper D

Industrial Procedures and Plant

2-5 p.m. on Friday, 30th May 1958

(SIX questions only to be attempted)

- 1. Starting with a loom-state cotton poplin, describe the processing routine you would use to produce a blue for shirting.
- 2. Describe a warp-dyeing machine. How would you vat-dye a cotton warp a medium brown using such a machine?
- 3. Give an account of the metachrome process for dyeing wool. What are its advantages and disadvantages compared with the chrome-mordant and afterchrome methods?
- 4. Why is wool dyed in the form of slubbing? Describe a machine with which you are familiar for dyeing slubbing—
 - (a) In hank
 - (b) In top.

What properties are required in dyes to be used on slubbing?

5. Describe a modern jig. What difficulties are encountered in obtaining uniformly dyed fabrics on this machine, and how can they be minimised? Illustrate your answers by reference to the dyeing of a secondary cellulose acetate poult.

- 6. How would you dye and finish two of the following warp-knitted fabrics for lingerie—
 - (a) Secondary cellulose acetate
 - (b) Cellulose triacetate
 - (c) Nylon?
- 7. Write an essay on one of the following topics—
 - (a) The use of reactive dyes in textile printing
 - (b) The use of resin-bonded pigments in textile printing
 - (c) Progress in the mechanisation of screen printing.
- 8. Describe the procedure you would adopt to dye Terylene slubbing with disperse dyes.
- 9. Discuss the relative merits in textile printing of application of colour by—
 - (a) Engraved roller
 - (b) Silk screen
- with reference to (i) cost and rate of production, (ii) ability to reproduce specific features of design.
- 10. What factors would you take into account in deciding whether a fabric should be winch-, jigger-, or pad-dyed?
- 11. Give a short account of the methods that have been suggested for the continuous, or semi-continuous, dyeing of cellulosic fibres with vat and direct dyes.
- 12. Describe **two** of the following machines and state their advantages—
 - (a) Barotor machine
 - (b) Pad-Roll machine
 - (c) Burlington machine
 - (d) Pressure jigger
 - (e) Star frame.

Paper E

10 a.m.-1 p.m. on Saturday, 31st May 1958

(Either Paper (i) or Paper (ii) to be attempted)

(i) Analytical and Testing Techniques

(FIVE questions only to be attempted)

- 1. An unevenly dyed fabric may show the fault either (a) in irregular areas or (b) in sharply defined areas extending the whole width of the fabric or garment. Draw up a scheme of examination for determining the reason for the unlevel dyeing in each of these cases. Give some indication of the principles underlying the tests applied and the results anticipated.
 - 2. Explain how-
 - (a) The load-extension curve of wool fibres may be determined
 - (b) The supercontraction of wool fibres is measured.

Discuss the results which might be expected in the case of wool fibres previously treated with

- (i) sodium nitrite in glacial acetic acid, (ii) sodium bisulphite, (iii) potassium cyanide, (iv) benzoquinone.
- 3. Explain the principles underlying fluidity determinations on textile fibres. Briefly outline the essential differences in the procedure for measuring the fluidity of—
 - (a) Viscose rayon
 - (b) Silk
 - (c) Nylon
 - (d) Cotton.
- 4. Write brief explanatory notes on any three of the following—
 - (a) Congo Red test
 - (b) Copper number
 - (c) Alkali-solubility test
 - (d) K.M.V. test.
- 5. Explain the general principles and methods used in the preparation of textile fibres for microscopic examination to reveal—
 - (a) Normal fibre outline and general surface appearance
 - (b) Medulla and other internal features
 - (c) Appearance in cross-section
 - (d) Surface structure by casts and impressions.
- Outline the tests which would have to be applied to dyed fabrics intended for bathing costumes.
- 7. Discuss the efficacy of the methods so far proposed for the determination of the fastness of dyed fabrics to gas-fume fading.
- 8. Outline the tests required for, and explain the principles underlying, the determination on a sample of dyed fabric of (a) the chemical type of the dye or dyes and the dyeing class, (b) whether the sample has been dyed with a single dye or a mixture.
- 9. Explain the method and principles involved in—
 - (a) The chromatographic separation of amino acids
 - (b) The chromatographic analysis of dyes from a dyed fabric.
- 10. Give an account of a satisfactory method for the quantitative determination of any two of the following—
 - (a) Organic isocyanates
 - (b) Methoxyl groups
 - (c) Polyhydrie alcohols
 - (d) Epoxides
 - (e) Amino acids.
- 11. A dyeing fault is believed to be caused by oil residues on the fabric. Explain what steps would be taken to test this assumption, and give some idea of the results which would be obtained in a particular case.

flammability of textile fabries and the kind of employees. Discuss the modern approach. results obtained.

(ii) Organisation of Production and Economics

(FIVE questions only to be attempted)

- "The evolution of industrial organisation has developed the foreman's position from that of leading craftsman to that of supervisor of his department's activities." Discuss this statement.
- 2. How would you organise a medium-sized bleaching, dyeing, or printing works?
- 3. What do you understand by joint consultation?
- 4. Give a short survey of the evolution of the personnel function in a works, i.e. matters relating

12. Discuss the methods of assessing the to the employment, welfare, health, and safety of

- 5. Discuss briefly two wage-incentive methods.
- 6. What is the function of the costing department and what are the main items of cost to be considered in calculating the total cost of bleaching, dyeing, or printing?
- 7. What do you understand by time and motion study?
 - 8. Discuss stock control.
- 9. "The day of the small processing works is over." Examine and criticise this statement.
- "The bleaching, dyeing, and printing trade is concerned with selling a service. It has no direct contact with the final users, and there is little that it can do to influence or to fix the scale of operations, i.e. the quantity of work." Discuss.

Report of the Examinations Board

The 1958 Examination was held in Belfast, Bradford, Nottingham, and Paisley on 29th, 30th, and 31st May 1958. There were 40 candidates, of whom 16 took only the first part of the examination (two of them took Papers A, B, and C, under the terms of the revised regulations), 6 the second part only, and 18 the examination as a whole. All those taking only the second part of the examination or the examination as a whole did so in Branch 1— Tinctorial Technology as applied to Textiles.

Of the 16 candidates who took only the first part of the examination 8 satisfied the Board, as did 4 of the 6 who took the second part only. Of those who took the examination as a whole 11 satisfied the Board, 6 were deemed to have failed, and one did not complete the examination.

As a result of the examination, which, it is stressed, includes the interview as well as the written papers, 15 candidates are recommended for election as Associates, their names being given in the Appendix*.

General Comments

In the written papers far too many candidates showed inability to express themselves adequately. From their papers and the interviews it is clear that few of them had had sufficient practice in writing essays or in answering typical examination questions with subsequent critical examination of their essays or answers by their instructors or other competent persons. Inability to express himself adequately is a serious failing in a candidate, for an Associate of the Society should not only have knowledge but be able to convey it to others; above all, he should be able to write concise, intelligible reports on any problem submitted to him in his professional capacity.

Handwriting was more legible this year, there being very few cases where difficulty was experienced in deciphering the answers.

Still a few candidates showed that they had not appreciated the standard required and so took the examination without sufficient preparation. It must be said that the average candidate did not reach the standard shown in previous years. This is seen not only in the decrease in the proportion of candidates who satisfied the Board but also in the quality of the papers presented by the satisfactory

A very few candidates had not realised that this is an examination in tinctorial technology and that, however much knowledge they could display of other subjects, their limited knowledge of matters relating to the application of colorants and the properties of coloured materials would make it inevitable that they could not satisfy the Board of their competence in tinctorial technology. A candidate may be highly skilled in matters relating to the manufacture of a particular substrate but have only an extremely limited knowledge of the application of colorants to that substrate. Such a candidate should take steps both to widen his knowledge of the use of colorants on the substrate with which he is concerned in his daily work and to obtain a general idea of industrial procedures in the application of colour to other substrates dealt with in the branch of tinctorial technology in which he asks to be examined.

The interviews again proved to be an invaluable help to the Board in assessing the merits of the candidates. In addition to interviewing the candidates who took either the whole or the second part of the examination, two prospective candidates about to go abroad, who intend taking the examination overseas next year, were also seen by the Board.

The following are the Board's and the Examiners' comments on the written papers-

Paper A

This paper was taken by 33 candidates, 23 of whom satisfied the examiners. In general the answers to Section I were not of high quality, and

many candidates betrayed lack of the necessary fundamental knowledge of chemistry. It would seem that a number of candidates had not preserved proper balance in their preparation for this paper, and had concentrated on the chemistry of dyes and neglected that of their intermediates. Candidates were better prepared for Section II, and in a fair number of cases it was their good showing in this section in spite of their weaker efforts in Section I that enabled the Board to regard their papers as satisfactory. There was no case where a good effort in Section I was deemed to compensate for weakness in Section II. The examiners comment as follows—

SECTION I

In general the standard of answers was quite good, being slightly lower than that of 1957. Questions answered best were those relating to the uses and mode of action of auxiliaries or to the properties of dyes. Weak answers were given to those questions concerning chemical constitution and synthesis. Few candidates answered Questions 7 and 8.

SECTION II

As in the previous year there were few outstanding papers, but most candidates showed reasonable grasp of the subject. It is encouraging to note a better grasp of physicochemical principles and less preoccupation with material solely of textile interest.

Few candidates attempting Question 9 could give methods of cross-linking, although the conditions for cross-linking of condensation polymers and the effects of cross-linking were often understood. Few candidates attempted Question 10, asking for distinction between atactic, isotactic, and syndiotactic polymers. Stereospecific polymers have been known for some years, and should be included in a candidate's course of study. The terms cationic polymerisation, block polymer, and emulsion polymerisation were generally understood, but few candidates knew what was meant by an azeotropic copolymer. There were very few good accounts of the structure of urea-formaldehyde resins in answer to Question 12, many being little more than an account of their preparation. In contrast, a few accounts of chain folding in proteins were quite good. Most candidates attempting Question 13 had reasonable understanding of how molecular weights could be obtained from measure-ments of osmotic pressures. In many cases, however, the description of the apparatus used suggested that some candidates had not included them in their course of study. A course in preparation for this paper should include at least consideration and, where possible, demonstrations of methods and apparatus used in the study of the characteristics of high polymers. Essays, again, were often not well ordered or balanced accounts of the topic chosen. In some cases, essays on vinyl polymers were essentially only accounts of the polymerisation of vinyl compounds. However, those on polyamides, the most popular topic, did generally include some account of structure and properties as well as preparation.

Paper B

This paper was admittedly difficult, but even so the standard of replies to it remains at a low level. Nevertheless, that 18 of the 33 candidates taking the paper should be deemed to qualify seems to be reasonably satisfactory.

The examiner's detailed report on the answers is—

QUESTION 1 (60%*)—In general, the candidates did not answer the question asking them to describe the principles of a spectrophotometer but tended to

· Proportion of candidates answering the question.

give a factual description of a particular instrument. Only one or two candidates had any idea of how to verify that the optical density scale of an instrument is correct.

QUESTION 2 (70%)—In this question candidates fell into two distinct classes—namely those who gave a clear and excellent answer, and the rest, who appeared to have little idea of the correct method of approach. Very few candidates seemed to appreciate the simplification possible as a result of the fact that the mixture curve is identical with the curve of dye B above 600 m μ . and therefore the mixture must contain 1 g. of B per litre.

QUESTION 3 (57%)—In general the candidates made a reasonable attempt at this question, but very few were able to describe a practical method of spacing patterns uniformly. The spinning disc method of mixing colours to obtain the shade of an intermediate colour appears to be almost unknown.

QUESTION 4 (23%)—The answers to this question were of a poor standard. All candidates mentioned the use of brightening agents, but other examples, such as camouflage, fluorescent paints and garments, heat reflection and solar evaporation of salt solutions, were rarely mentioned.

QUESTION 5 (20%)—Only a small number of candidates answered this question, which suggests that students do not take a sufficient interest in papers published in the Journal, as the question was really a simple descriptive question. The majority of candidates concentrated on the use of colorimetry to follow the reduction of vat dye solutions, and the answers were of a good standard.

QUESTION 6 (37%)—The replies to this question were of very variable quality.

QUESTION 7 (53%)—Most candidates made a fair attempt at this question, but failed badly on the last section, namely on ensuring that the colour specification could be met in bulk production.

QUESTION 8 (70%)—A relatively simple question attempted by a large number of candidates, who, in general, showed a good grasp of the subject but often presented their ideas badly.

QUESTION 9 (23%)—Few candidates attempted this question, and the answers, although usually lengthy, were not of good quality, indicating that the subject of colour and constitution is still neglected by students.

QUESTION 10 (10%)—This was a difficult question and evoked only a few gallant attempts. Again, the answers to the last section indicate that disc mixing is unknown.

Question 11 (32%)—Few candidates had a real understanding of the meaning of standard observer. Nearly all answered the question on light sources correctly and in greater detail than expected, but there was surprising ignorance of standard viewing conditions, only two candidates mentioning the limitation of the field of view to 2° and the illumination of the pattern at 45° coupled with viewing at 90°.

QUESTION 12 (45%)—Again a relatively simple question which produced some good answers, and also one or two very poor answers in which the wool-nylon fabrics were dyed quite separately.

BRANCH 1— TINCTORIAL TECHNOLOGY AS APPLIED TO TEXTILES

Paper C

Of the 26 candidates who took this paper 24 were deemed to qualify.

The content of the paper was somewhat modified this year to bring it more closely in accord with the actual syllabus and to make it deal more definitely with the theoretical aspects of textile

dyeing and printing. The marking was therefore a little more lenient. The total result was not quite satisfactory. There were no abject failures, but the best performances were not remarkable. The chief cause of this has been mentioned in earlier years, viz. that candidates do not yet prepare themselves sufficiently thoroughly for quite moderate demands on their theoretical knowledge and rely too much on their practical experience-often considerable-to see them through. Provision is made in other papers in this examination for them to display this experience, and again it should be realised that Associates who expect to handle the tinctorial processes of the future will not be able to rely wholly on their practical experience. Many times in this paper critical comment was specifically invited but was not forthcoming, and the impression was gained that candidates had had very little previous experience of tackling questions of this type. Further, they would be well advised to give a little more thought to each question before committing themselves to paper. Many answers would then be shorter, less diffuse, and more to the point. This, of course, holds for the other papers too.

The examiners' report on the individual questions, the number of attempts at each being given in parentheses, is—

QUESTION 1— Part (i) (2)—The candidates who answered this question had not read very widely, and those who had done so tended to comment on the method of presentation rather than on the content of the work they discussed.

Part (ii) (8)— Comment as for Part (i).

QUESTION 2— Part (i) (20)— A popular question with a good proportion of good to very good answers. Very few candidates mentioned either the Abbot-Cox process or the vat-acid process.

Part (ii) (4)— Most references were to the work of the ICI Dyehouse Department. There was some vagueness about the significance of the relation between the potential of the dye system and the reduction potential of the reducing agent.

QUESTION 3 (23)—A very popular question, but there was too much evidence of lack of real appreciation of the facts.

QUESTION 4 (15)—Too many incomplete answers. The formula of the monomer was not always included. Only a few candidates saw fit to include such a fibre as Acrilan under the general heading of polyacrylonitrile, and the application of basic dyes to these fibres was occasionally omitted.

QUESTION 5 (9)—With one or two exceptions the answers were very poor. Fundamental knowledge of the textile chemistry of wool seems sadly lacking, as does that of the dyeing behaviour of modified wool.

QUESTION 6 (13)—The inaccuracy of much of the knowledge about the reactions of cellulose was rather shocking. Hydrolysis was confused with oxidation; the constitution of anhydroglucopyranose was not known; the consequences of periodate oxidation were not understood; and while the candidates appreciated that oxidation under acid and under alkaline conditions gives different results, they were not clear why this is so.

QUESTION 7 (18)—Some quite good answers. The possibility of covalent dye-fibre bonds passed almost unnoticed. The relations between bond strength, the migrating power of the dye, and the wet fastness of dyeings were not always clearly brought out. There was a great need of more systematic presentation.

QUESTION 8 (1)— Much of the detail in the answer was inaccurate.

QUESTION 9 (2)—One candidate did not define the difference between Newtonian and anomalous viscosity very clearly, although he tried hard. The other had never heard of the work and was scornful, as a practical printer, about the utility of quantitative definition of printing quality. He put in a very entertaining argument and was given credit for this.

QUESTION 10 (12)—The existence of a possible hydrazo stage in the reduction of azo dyes was generally ignored. Not many considered the fate of the reduction products after the azo group had been attacked. Only one candidate mentioned the Leucotrope reaction in vat discharge printing.

QUESTION 11 (19)—Examples of the utility of pH measurement were many, and some were imaginative. However, probably because pH-meters have become "black boxes", incidental remarks revealed agreat deal of ignorance of the elementary principles of the glass electrode and the valve electrometer.

QUESTION 12 (9)—Knowledge of simple dyeing theory is still very shaky.

Paper D

Of the 24 candidates who took this paper 18 were deemed to qualify. As the examiner comments (see below) the knowledge of some candidates was limited to very narrow fields. It is appreciated that few candidates can have had wide practical experience, but every candidate can read about branches of dyeing or printing other than the one he is engaged in and can discuss them with other persons; indeed, it is on these very foundations that the Society is based. Many firms would be willing to allow non-competitors at least to see their works on a reciprocal basis. Lack of any thought of, or effort toward, doing these things is an even more serious defect in a candidate than is the actual lack of knowledge of fields outside his own.

In his report the examiner states that-

In general the questions set for this part of the examination have been answered at a reasonably high standard. One noticeable feature, however, is the wide variation in the standard of answers in the same paper to different questions. This points to candidates having specialised knowledge in one particular field of the industry, rather than having a general knowledge of dyeing such as is required by an examination of this type.

As in previous years, there is again some evidence of inadequate preparation for the examination. This, together with the noticeable concentration on the requirements of local sections of the dyeing industry, suggests that the teaching curriculum in some of the courses leading up to this examination cannot be wholly satisfactory. This specialisation is sufficiently obvious for one to feel it possible to tell, from the quality of the answers given, the district where the examinee has been trained.

The old failure of tending not to read the question, or of hoping to achieve marks by answering only part of the question, is again noticeable. Thus, in Question 6, on nylon warp-knitted fabrics, one examinee answered the question with reference to nylon stockings, whilst in Question 1 another examinee dealt with the preparation and bleaching of a cotton poplin without any reference to how to dye the fabric blue, as asked for in the question.

Some of the terminology used was, to say the least, peculiar. The term "artificial silk" instead of rayon is bad enough, but why use the term "artificial rayon"?

One disturbing feature in many cases is the lack of basic knowledge. Thus the following statements were made by examinees—

Question 5— "The exposed selvedge will tend to dye darker due to exposure to light when dyeing with dispersed dyes."

Question 11—"The hot oil process is one which uses the violent reaction between hot oil and water where heat is liberated."

Both these statements are, of course, not only incorrect but ludicrous.

As in the 1957 examination, jigger dyeing is again suggested for dyeing warp-knitted fabrics, and attention was called to this in the examiner's

Those candidates who attempted this paper had either (a) received a wide course of instruction or (b) had sound experience in the application of certain methods. In general it could be assumed that those in category (a) had tended to forget the minute details originally acquired in courses of instruction, and had not sufficiently replaced this knowledge of detail of method by appreciation of implication of the results, or of the reasons for the tests. It is still felt that far too few candidates really understand the implication of many of the tests, and cannot adequately discuss the results with respect to the end-product.

The average marking obtained was 60%, and for individual questions was—

											Dinner		
Question No.		1	2	3	4	5	6	7	8	9	10	11	12
No. of answers										2	0	6	6
Average mark	004	15	13	13	10	11-5	11	16	12.5	11	-	8	12

comments published following the last examination (J.S.D.C., 73, 454 (1957)).

Paper E(i)

Seventeen candidates took this paper, of whom 15 proved satisfactory. It is a matter of surprise to the Board that so many candidates who do no analysis or testing in the course of their normal work should prefer to take this paper rather than Paper E(ii), for when the original pattern of the Associateship examination was devised, Paper E(i) was intended for candidates engaged in laboratory work, testing, research, or quality control, and Paper E(ii) for those actually engaged in production. It would seem that the fact that formal courses in this subject are readily available in schools of dyeing but none in that covered by Paper E(ii) has largely deterred those who in fact are better fitted to take Paper E(ii) from taking it instead of Paper E(i). That most of those taking this paper can reach the necessary standard is a tribute to the training given in this subject. The examiner reports that-

There was a very distinct improvement in the standard of answers compared with the previous years.

It is noteworthy that the most popular question (No. 4), on specific tests, was the worst answered, for the very reasons detailed above. No candidate attempted Question 10, on analysis of organic materials, and only two answered Question 9, on special methods of analysis.

Paper E(ii)

Five candidates took this paper, the same number as last year. Two returned very good papers, another a fairly good one, and the other two just managed to qualify. No candidate had taken a formal course in preparation for this paper, which makes the standard reached by three of the candidates all the more praiseworthy and should encourage future candidates engaged in production work to take this paper. The examiner comments that the lack of any formal preparation was noticeable in the replies given to Questions 2 and 5 and in Question 4 not being attempted. It was pleasing that candidates showed improvement in the economic appreciation of the subject.

C. O. CLARK (Chairman)

R. L. ELLIOTT	J. V. SUMMERSGILL
W. Penn	H. A. TURNER
C. B. STEVENS	T. VICKERSTAFF

Proceedings of the Society

The Dyeing of Acrilan Acrylic Fibre

H. R. HADFIELD and W. M. SOROL

Meetings of the West Riding Section held at the Victoria Hotel, Bradford, on 31st October 1957, Mr. G. E. Styan in the chair; and of the Scottish Section held at the St. Enoch Hotel, Glasgow, on 10th December 1957, Mr. W. G. B. Grant in the chair

The methods of dyeing Acrilan are outlined, and the limitations of various classes of dyes stated. Data are presented to show how the concentration of sulphuric acid affects the handle of the fibre and the fastness properties of acid, chrome, and 2:1 metal-complex dyes. Generally, disperse dyes are the most satisfactory for the dyeing of Acrilan, although for specialised purposes azoic, acid, basic, chrome, metal-complex, and reactive dyes are suggested.

INTRODUCTION

Since the introduction of Orlon acrylic fibre by du Pont, it has been recognised that, for many purposes, difficulties in exploiting the desirable textile characteristics of acrylic fibres arise from their dyeing properties (although recently the introduction of completely new types of basic dyes 1 has done much to eliminate this disadvantage). The Chemstrand Corporation realised the difficulty associated with the dyeing of acrylic fibres and introduced an acrylic fibre modified with basic molecules, to confer on the acrylic fibre affinity for anionic dyes. Almost an incidental result of this modification was an improvement in the dyeing properties for disperse dyes.

In the early days of the technical appraisal of Acrilan in the dyeing industry, attention was concentrated upon the dyeing properties of acid, metal-complex, and chrome dyes 2,3, and emphasis was laid on the "wool-dyeing" behaviour of the fibre. During the last twelve months, considerable quantities of Acrilan have been processed by the textile industry in Britain, and this has enabled the dyeing techniques, initially formulated in the laboratory, to be reviewed in the light of practical experience. This experience is summarised below.

(1) Acid Dyes

Acid dyes were found to be very difficult to apply level to yarn and cloth, and the high quantities of acid required usually had a detrimental effect upon the desirable handle of Acrilan. Some difficulty was also experienced in producing level results on loose fibre and slubbing, particularly when applying ternary mixtures of dyes. The difficulty of applying ternary mixtures to Acrilan is accentuated by the need to use mixtures of level-dyeing acid dyes and acid milling dyes in the presence of high concentrations of sulphuric acid in the dyebath, in order to obtain adequate fastness. Blue acid dyes of high fastness to light are not generally obtainable. Nevertheless, in spite of the difficulties referred to above, acid dyes are used, with satisfaction, for certain purposes.

(2) Acid Levelling Metal-complex Dyes

High concentrations of sulphuric acid in the dyebath are required, and the levelling properties of acid levelling metal-complex dyes are poor. The absence of a fast-to-light blue dye for use in pale and medium depths is a serious limitation. Ultralan dyes are, however, used with satisfaction for certain heavy shades, such as bottle greens, on loose fibre or slubbing. Again the effect on the handle of the fibre caused by high concentrations of sulphuric acid in the dyebath constitutes a serious disadvantage.

(3) Neutral-dyeing Metal-complex Dyes

These dyes are not as satisfactory on Acrilan as on wool. Difficulties are caused by their lower fastness to wet treatments, poor building-up properties, poor penetration of the fibre, and possible demetallisation by the sulphuric acid used in application. Nevertheless, for the production of fast pale colours on loose fibre or slubbing they are of considerable value if dyes are carefully selected.

(4) Mordant Dyes

Mordant dyes are of no value for yarn or cloth, but, in very heavy depths, selected chrome mordant dyes can be used to dye loose fibre or slubbing with excellent fastness. An afterchrome process is essential, but there are difficulties in application similar to those found with acid dyes.

(5) Reactive Dyes

The present range of Procion and Procion H dyes is not generally of value for dyeing Acrilan. The excellent wet fastness obtained would be very attractive for the slubbing or loose-fibre dyer, but red dyes of high fastness to light are not yet available. In addition, for certain purposes the poor levelling properties of these dyes on Aerilan constitute a serious disadvantage.

(6) Basic Dyes

The use of basic dyes is limited to those cases where, from shade considerations, no alternative is possible.

With the above experience it was decided to re-examine the various classes of dyes, compare the fastness properties obtained, and then assess the results, bearing in mind the relative ease of application of each class.

COMPARISON OF FASTNESS PROPERTIES

A pale, a medium, and a heavy grey were dyed on Acrilan yarn using the optimum dyeing conditions with the following classes of dyes— (a) disperse

TABLE I

				Compa	Comparison of Fastness on Acrilan	ness on Acril	an an				
Type of Dye	Depth (relative to standard)	Light	S.D.C. W Effect on Colour	S.D.C. Washing Test No. 2 Effect on Staining of Colour Acrilan V	fo. 2 ing of Wool	Effect on Colour	Perspiration I* Staining of Acrilan Wo	ing of Wool	Effect on Colour	Alkaline Milling* Staining of Acrilan W	ng of Wool
Disperse	-	10	10	50	10	.0		10	10	10	10
	1	9	2	10	4-5	10	9	4-5	2	5	10
	03	5 Redder	4-5	13	3 Bluer	10	10	3-4 Bluer	10	10	3-4 Blue
Acid	e-jai	3-4 Bluer	10	10	10	10	10	10	10	9	10
	-	4-5	10	10	10	10	4-5	4-68	10	9	15
	09	10	9	10	4-5	10	. 4-5	4-0	10	9-9	4-3
Chrome	origin .	*	10	10	9	10	10	10	10	10	10
		4-5	10	10	10	10	10	10	10	10	10
	63	2	2	5	2	5	20	10	10	5	10
2: 1 Metal-	-4n	9	50	10	10	20	4-5	4-5	22	4	22
complex	1	9	4 Redder	+	2	20	4-5	4-5	*	*	10
	45	4 Greener	4 Redder	ಣ	4-5	4	01	3.	4	69	10

† Dyed with 6% sulphuric acid.

TABLE II

Wool Wool Bluer

dyes, (b) acid dyes, (c) chrome mordant dyes, (d) 2:1 metal-complex dyes. In all cases, except where indicated, the preferred dyes were employed. The dyeings were then tested for fastness to various agencies.

An examination of the results in Table I shows that in all cases, with the disperse, acid, and chrome dyes applied in pale and medium depths, excellent wet fastness is realised. With medium depths of the 2:1 metal-complex dyes there is some staining of adjacent whites. The tests on heavy dyeings are also of interest: the high wet fastness is maintained with the acid and chrome dyes, but serious staining of adjacent whites is found with the greys produced from disperse and metal-complex dyes. In addition, the dyeing obtained with double the amounts of metalcomplex dyes required to produce the standard depth shows little visual increase in depth. The question of build-up with metal-complex dyes will, however, be discussed in detail at a later stage.

It is known, from results which will be described later, that the stain on adjacent whites in wetfastness tests on the heavy depth obtained with disperse dyes is due to the use of a blue dye with poor building-up properties, which thus tends to be deposited loosely on the fibre surface. Disperse dyes can, however, be used to produce heavy depths of good fastness, provided that dyes of good building-up properties are selected. In this particular case, Duranol Blue G (C.I. Disperse Blue 26) was used as a general blue component for the pale, medium, and heavy depths. The advantageous effect on wet fastness of replacing it, in a recipe to produce a deep grey, by Duranol Brilliant Blue CB (C.I. Disperse Blue 1), which has good build-up on Acrilan, is seen from Table II.

DISPERSE DYES

The preliminary results suggest the value of a fuller examination of the behaviour of disperse dyes on Acrilan than has yet been described. A beginning was made by measuring the saturation values of a series of disperse dyes on Acrilan, and a comparison was made with the same dyes applied to Orlon 42 and to secondary cellulose acetate. It will be seen (Table III) that both acrylic fibres possess lower saturation values with disperse dyes compared with cellulose acetate, and that, with the exception of Duranol Blue G, Acrilan possesses a higher saturation with the disperse dyes examined than does Orlon 42.

TABLE III Saturation Values for Disperse Dyes on Acrilan (100% Dye at 98°c. for 48 hr.)

Saturation Uptake

	(g. dye Acrilan	per 100 Orlon 4	g. fibre) 2 Cellulos Acetate
Dispersol Fast Yellow G 300 (C.I. Disperse Yellow 3)	2-6	1.3	7-4
Dispersol Fast Orange B 300	6-0	2.8	10-0
Duranol Red X3B 300	7-9	7-6	12.2
Duranol Blue G 300	3-2	4.0	8.6
Duranol Brilliant Blue CB 300	8-3	8.0	10-6

In using the results in Table III, it must be emphasised that the saturation values were obtained by dyeing with very high concentrations of dye under conditions allowing equilibrium exhaustion to be attained. The results obtained for the saturation values on Orlon 42 do not agree closely with those obtained by Walls 4. It is probable that the slightly higher saturation values obtained in this work result from a slightly higher dyeing temperature. These conditions are remote from the dyeing conditions generally used in practice, and practical dyeing tests indicate that the differences in dyeability with disperse dyes between Orlon 42 and Acrilan are greater than the saturation values would suggest. For example, Fig. 1 and 2 show the building-up properties on Acrilan and Orlon 42 of Dispersol Fast Orange B (C.I. Disperse Orange 13) and Duranol Brilliant Blue CB under practical dyeing conditions.

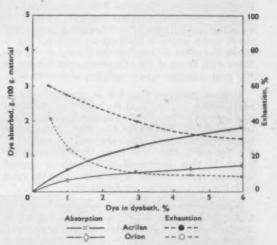


Fig. 1— Building-up Properties of Dispersol Fast Orange B (C.I. Disperse Orange 13) (dyed with 1/1000 Lisaspol C paste in 40: 1 liquor at 98°c. for 90 min.)

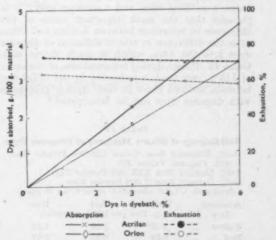


Fig. 2— Building-up Properties of Duranol Brilliant Blue CB (C.I. Disperse Blue 1) (dyed with 1/1000 Lissapol C paste in 40:1 liquor at 98°C, for 90 min.

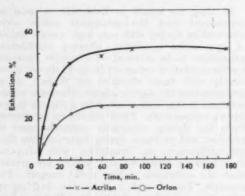


Fig. 3— Rate of Dyeing of 2-0% Duranoi Blue G 300 Powder Fine (C.I. Disperse Blue 26) (dyed with 1/1000 Lissapol C in 40; 1 liquor at $98^\circ\mathrm{C.}$)

It will be seen that Dispersol Fast Orange B, which may be regarded as a typical disperse dye in so far as behaviour on Orlon 42 and Acrilan is concerned, shows much superior build-up on Acrilan. Fig. 3 shows the effect of time on the dyeing of the two fibres with Duranol Blue G, which dyes Acrilan at 100°c. at a similar rate to cellulose acetate at 85°c.

Duranol Brilliant Blue CB is essentially tetraaminoanthraquinone, and its behaviour on Orlon 42 is exceptional in that it possesses good buildingup properties. The explanation of its exceptional behaviour may lie in the very basic character of the dye (it is the most basic of disperse dyes), which is thus intermediate between a typical disperse dye and basic dyes which possess good building-up properties on Orlon 42.

A further good example of the improvement in building-up properties of disperse dyes applied to Acrilan compared with Orlon 42 is illustrated in Fig. 4. A maroon, a brown, a royal blue, and a black have been obtained with disperse dyes on Acrilan; with the same dyeing conditions, the same recipes applied to Orlon 42 produced a pink, a beige, a medium blue, and a medium grey. It is thought that the most important cause of the difference in behaviour between Acrilan and Orlon 42 is the difference in rate of diffusion of disperse dyes into the fibre, which is much slower with Orlon 42. If high dyeing temperatures, of the order of 115°c., are used, a reduction in the difference between the two fibres in their dyeing properties with disperse dyes can be anticipated

TABLE IV

Building-up of Binary Mixtures of Disperse Dyes

(5.0% Dispersol Fast Yellow GR 300 Powder Fine (C.I. Disperse Yellow 39)

5.0% Duranol Red X3B 300 Powder Fine 5.0% Duranol Blue G 300 Powder Fine

5.0% Duranol Blue G 300 Powder Fine dyed with 1/1,000 Lissapol C at 98°c. for 90 min.)

Admixed	d	Yellow (g. Dye per	Red 100 g. m	Blue aterial)
Yellow		1.58*	2.19	1.13
Red	440	1.68	2.23	1.17
Blue	***	1.60	2.08	1.11

[•] Figures in italics refer to the individual dyes (5%).

In spite of the superior building-up properties on Acrilan compared with Orlon 42, difficulty is still experienced with homogeneous dyes when attempting to produce full colours. It is known, however, that on cellulose acetate and Terylene mixtures of dyes build up independently 6-8. Obviously this principle should apply also to Acrilan. Tests have been carried out by applying a yellow, a red, and a blue dye to Acrilan, measuring the dye absorbed, and then repeating the measurements using the dyes in admixture. The results in Table IV clearly illustrate that the dyes selected do in fact build-up on Acrilan independently of each other. It is to be emphasised that the practical dyer would be wise to adopt this principle for producing heavy depths, as, not only is there an economic advantage, but there is less danger of dye being deposited on the fibre surface as a result of "oversaturation". illustration of the value of using mixtures is provided in Fig. 5, showing 0.5%, 1%, 2%, 4%, and 8% dyeings of Duranol Blue G 300 and Duranol Navy Blue BN, which is a mixture: the homogeneous dye ceases to build up at approx. 2%, whilst Duranol Navy Blue BN is still building up at an 8% dyeing.

TABLE V

Levelling Properties of Disperse Dyes on Acrilan (Fresh bath containing 1/1,000 Lissapol C at 95°c. for 1 hr., liquor ratio 40:1)

Dyes	Levelli	ing (%)
and the original property in the first	95°c.	100°c.
3.0% Duranol Red 2B 300 Powder Fine (C.I. Disperse Red 15)	69-4	74-1
3.0% Dispersol Fast Scarlet B 150 Powder Fine (C.I. Disperso Red 1)	20-9	51.9

Another very important property of disperse dyes is their ability to level. This property is of the greatest importance in the dyeing of piece goods and yarns, and it is important also in the package dyeing of acrylic fibres, as in hot dye liquors acrylic-fibre packages become very compact, resulting in a low flow of dye liquor and thereby a frequently uneven initial absorption of dye. Table V shows that good levelling is obtained with disperse dyes on Acrilan, but that the effect is very dependent upon the temperature. This is particularly the case with the moderately or slowly diffusing dyes, such as Dispersol Fast Scarlet B (C.I. Disperse Red 1), but is not marked with a rapidly diffusing dye, e.g. Duranol Red 2B (C.I. Disperse Red 15).

It must be emphasised that, in spite of the good levelling properties of disperse dyes on Acrilan at 95–100°c., these dyes possess good fastness to wet treatments at 60°c. or below. It is thought that this fortunate behaviour is a result of a very high activation energy of dyeing, such as is found with Terylene 9, and contrasts with that of direct dyes on viscose rayon. The wet fastness of disperse dyes applied to Acrilan is higher than that found for disperse dyes applied to nylon and cellulose acetate rayon, and is of the same order as found for disperse dyes applied to Terylene and cellulose triacetate 4.

4-8% Dispersol Fast Yellow GR 300 Powder Fine (C.I. Disperse Yellow 39)

1-2% Duranol Red X3B 300 Powder Fine (C.I. Disperse Red II)

0-6% Duranol Blue G 300 Powder Fine (C.I. Disperse Blue 26)

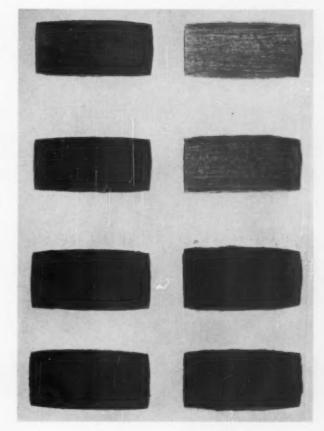
9-0% Dispersol Fast Yellow GR 300 Powder Fine (C.1. Disperse Yellow 39) 3-3% Duranol Red X3B 300 Powder Fine (C.1. Disperse Red 11)

2-4% Duranol Blue G 300 Powder Fine (C.I. Disperse Blue 26)

12:0% Duranol Navy Blue BN

BBBB , VI

15-0% Duranol Black BR 1-0% Dispersol Fast Crimson B 150 Powder Fine (C.I. Disperse Red 13)



Acrilan

Orlon

Fig. 4— Building-up of Disperse Dyes on Acrilan and on Orion (dyebath containing 1/1000 Lissapol C at 98° C, for 90 min.; soaped in 2/1000 Lissapol NC at 60° C, for 15 min.; liquor ratio 40:1)

Duranol Blue G 300 Powder Fine (C.I. Disperse Blue 26)

Duranol Navy Blue BN

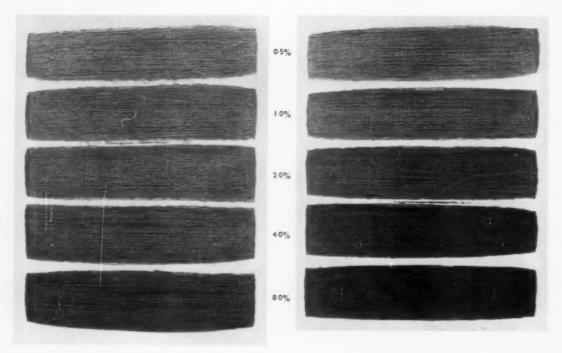
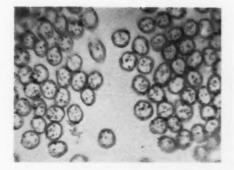
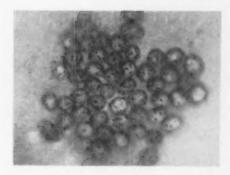


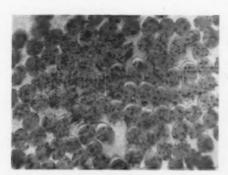
Fig.~5 - Building-ap~Properties~of~Disperse~Dyes~on~Aerilan~(dyed~with~1/1000~Lissapol~C~at~98°c.~for~90~min.;~liquor~ratio~40~;~1)



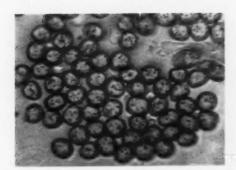
3% Sulphuric Acid, dyed for 1 hr.



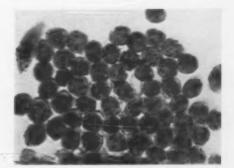
6% Sulphuric Acid, dyed for I hr.



10% Sulphuric Acid, dyed for 1 hr.



3% Sulphuric Acid, dyed for 1 hr.



3% Sulphuric Acid, dyed for 7 hr.

Fig. 7— Effect of Concentration of Sulphuric Acid on Penetration of Acrilan by Solway Green G (C.I. Acid Green 25)

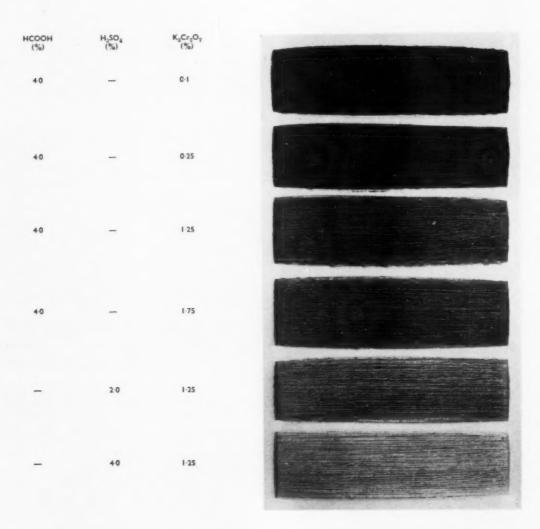


Fig. 9— Effect of Chroming Conditions on 3-0% Solochrome Red ERS (C.I. Mordant Red 7) on Acrilan (dyed with 6-0% sulphuric acid (168°Tw.) at 98°C. for 90 min.; chromed in a fresh bath at 98°C. for 90 min.; liquor ratio 40:1)

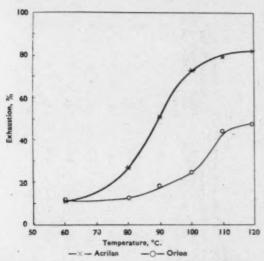


Fig. 6— High-temperature Dyeing of Acrilan and Orlon with 3% Dispersol Fast Scarlet B 150 (C.t. Disperse Red 1) (dyed with 1/1000 Lissapol C for 90 min.)

The high-temperature dyeing of Acrilan has been investigated, and the behaviour of disperse dyes above 100°c. is illustrated in Fig. 6. Increased exhaustion is obtained, when dyeing for 90 min., as the temperature is increased up to 120°c. The results also indicate that Orlon 42 possesses a temperature of maximum exhaustion of approx. 110°c. These results on Acrilan confirm those obtained by Kramrisch 5 with other disperse dyes. In considering the results in Fig. 6, it must be noted that Dispersol Fast Scarlet B is a dye which does not very readily diffuse into Acrilan or Orlon 42, and a wide range of alternative dyes are available which possess a lower temperature of maximum exhaustion and thus more readily build up to heavy depths. It is to be emphasised that at temperatures above 100°c. Acrilan is to some extent plastic in the dyebath, and for this reason high-temperature dyeing is not employed, nor is it generally necessary.

In selecting disperse dyes for Acrilan, besides the usual properties considered, such as light fastness, wet fastness, colour, and dyeing properties, the fastness to heat treatments is a most important property for many requirements, as Acrilan can be given durable pleating treatments by steaming. For this purpose a 10-min. treatment between atmospheric pressure and 5 lb./sq.in. steam pressure is used. Information on the heat fastness of disperse dyes to a pleating process has been compiled ¹⁰, and it will be seen that a sufficiently large range of dyes is available, fast to heat and other treatments, to enable disperse dyes to be used to dye all but heavy depths even on loose fibre and slubbing, where the highest standards are This conclusion is not generally essential. accepted 11, as in the past disperse dyes have been thought to be unsuitable for all but the palest of colours. High light fastness is generally difficult to achieve on Acrilan, but it is not more difficult with disperse dyes than with most other classes. Dyeing is generally carried out at the commercial

boil from a bath containing 1 part of Lissapoi D* per 1,000 parts of liquor, although, when dyeing bottle greens and dark blues, the Lissapol D can, with advantage, be replaced by Dispersol AC. In order that reproducible results can be obtained with disperse dyes, the dyer must ensure that the liquor ratio varies as little as possible between one dyeing and the repeat. It is important that, before commencing dyeing, the fibre should be free from finishing agent or antistatic or combing oils, as these can seriously affect the result. If these agents are present, scouring at 50°C with Lissapol D is necessary.

AZOIC DYES

Azoic dyes as a class are not of interest for the dyeing of Acrilan. The production of blacks, using Dispersol Diazo Black B (C.I. Disperse Black 1) and Brentosyn BB, Brenthol OT (C.I. Azoic Coupling Component 18), or Brenthol FR (C.I. Azoic Coupling Component 20), is an exception, in that very valuable results can be obtained with these azoic components. For most purposes, Brenthol OT is preferred to Brentosyn BB, as, with the former, both azoic base and coupling component may be applied to Acrilan simultaneously from the same bath 12. Azoic dyes are applied to Acrilan by techniques similar to those developed for the dyeing of cellulose acetate and Terylene, but the methods used to apply this class of dyes to cellulosic fibres are not suitable. In outline, the method consists in applying the Dispersol Diazo Black B and the dispersed Brenthol OT from a dyebath at 95-100°c., and subsequently diazotising in acidified sodium nitrite solution at 85°c. Full details of the method have been published 13, and the fastness properties indicate that the black, when correctly applied, is sufficiently fast to satisfy, for example, the rigorous requirements of the slubbing dyer. If desired, Brenthol FR can replace Brenthol OT, as it shows superior exhaustion at lower temperatures compared with Brenthol OT.

Three main methods of producing blacks on Acrilan have previously been suggested—

- Use of chrome dyes—Good fast blacks can be produced, but there are difficulties in obtaining reproducible results, and, in addition, the handle of the fibre is seriously affected.
- (2) Use of Ultralan Black RN (C.I. Acid Black 52)—Good blacks of moderate fastness are produced, but as large concentrations of sulphuric acid in the dyebath are essential, the handle of the fibre is seriously affected.
- (3) Use of Duranol Black 2BN—Good blacks of moderate fastness are produced, but the fastness is not sufficiently high for slubbing or loose-fibre dyers, and large amounts of dye are essential. Economic factors are therefore of primary importance.

Lisapol D (essentially sodium oleyl-cetyl sulphate) is normally used in practical dyeing, but in certain of the tests reported in this paper Lisapol C (essentially sodium cetyl sulphate) was used.

TABLE VI

	at 60°c. for 20 min.)	Allegias Milling
	for	Ilealie
	60°C.	A
	at	
	liquor)	
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	a as	
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Dye	1,00	- diam
Cid	+ 1	Dominion I
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	at 9	0
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	(168	
	e acid	T tombe
	(Dyed with 6% sulphuric acid (168°Tw.) at 98°c. for 90 min.; "soaped" in 2/1,000 Lissapol NC + 1/1,000 soda ash (40:1 lique	
	%9	
	with	
	Dyed v	Deres
	C	-

TABLE VII

Solubility and Fastness Properties of Selected Milling Acid Dyes (188°Tw.) in 40:1 liquor at 98°c, for 90 min.; "soaped" in 2/1,000 Lissapol NC + 1/1,000 soda ash at 60°c, for 20 min.)

TOTAL MODE	o de la	TOO TWILL	DI 1 : 04 II	101 car 30 c. 101	OO HIHIT.	and and	7 7 7000	A ALLESSON AND A	2 2	OOO OOO	CO 000 8500		1
Dye		Solul	bility*		Light	S.D.C. V	Vashing T	est No. 2	Pe	repiration	I	Alk	aline Milling
(medium depth)	5% (NH,),80,+	%2%	4%	2%	(day-	Effect	Staini	Jo Bu	Effect	Staini	Jo Bu	Effect	Staining
	2% НСООН(85%)	НСООЕ	(%98) I	H ₂ SO ₄ (168°Tw.)	light)	Colour	Acrilan	Wool	Colour	Acrilan	Wool	Colour	Acrilan
Coomassie Fast Yellow G (C.I. Acid Yellow 85)	aust Yellow G B† B B B 4	В	B	B	*	10	10	10	19	10	10	10	10
Carbolan Brown BL	В	B	B	Insol.	9	*	9	12	10	10	10	4-5	10
Connassie Fast Scarlet 2G (C.I. Acid Red 140)	Sol.		Sol.	Sol.	4	3 Yellower	10	10	10	4-5	2	*	Yellower 5 5 4-5 4-5 4 5
Coomassie Red G (C.I. Acid Red 116)	Insol.		Insol.	Insol.	*	10	10	10	*	3-6	80	*	10
Carbolan Blue B (C.I. Acid Blue 138)	Insol.		Insol.	Insol.	4	4-5	10	10	4-5	4-5	29-	10	19
	2R Sol.	Sol.	Sol.	Sol.	*	4	10	10	10	12	4-5	•	2
Coomassie Fast Grey 3G (C.I. Acid Black 48)	Insol.		Insol.	Insol.	9	4	4-5	4-5	4-5	4-5	4-5	+	*
Carbolan Brilliant Green 5G (C.I. Acid Green 28)	8G B	В	В	В	9		10	10	10	*	*	10	10

Water of 5° hardness at 55°C.; liquor ratio 10 : 1.
 ↑ B = Borderline.

It is suggested that the use of azoic dyes for the production of blacks offers a marked advantage, for most purposes, over the alternatives listed above.

For other colours azoic combinations do not generally offer advantages over other classes of dyes applicable to Acrilan, and, in addition, most azoic combinations giving scarlets, reds, and maroons, when applied to Acrilan, suffer from "blinding", i.e. migration of the azoic pigment to the fibre surface, when subjected to a steaming such as is used to give a permanent pleat.

ACID DYES

The wool dyer, on the basis of dyeing and fastness properties, has divided the acid dyes into two main groups—level-dyeing acid dyes and acid milling dyes. When these dyes are applied to Acrilan, this classification does not hold, as practically all acid dyes, including acid milling dyes, require sulphuric acid in the dyebath in

order to effect exhaustion. Furthermore, many instances are found where level-dyeing acid dyes applied to Acrilan possess very high wet fastness, which is superior to that obtained with certain acid milling dyes. In general, however, it is found that most acid dyes possess greater wet fastness on Acrilan, but lower light fastness, than on wool.

It has previously been shown that acid dyes show poor penetration of Acrilan unless the dyebath contains high quantities of sulphuric acid², although continuing the dyeing for a longer period also improves dye penetration. This effect is shown clearly by Fig. 7. Although the use of high quantities of sulphuric acid results in no difficulty in applying level-dyeing acid dyes, with the much less soluble acid milling dyes, unless care is taken, difficulty can be experienced resulting from dye precipitation.

Unfortunately, full advantage cannot be taken of the high wet fastness of the level-dyeing acid

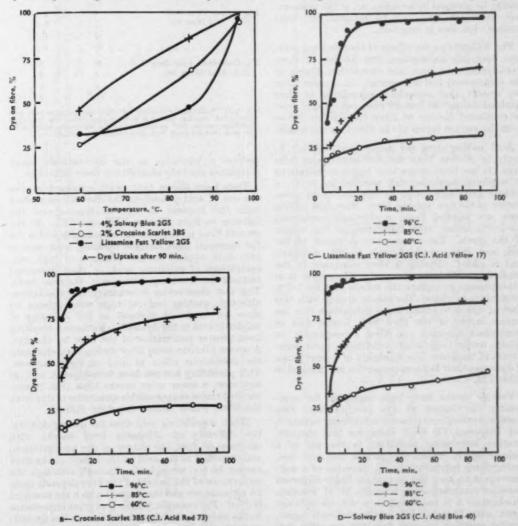


Fig. 8— Effect of Temperature on Dye Uptake and Rate of Dyeing of Acrilan (dyed with 4% sulphuric acid in 40;1 liquor)

dyes because of the absence of a complete range, including a yellow, a red, and a blue dye, which possesses satisfactory fastness properties for many requirements, particularly light fastness. In general, good yellow dyes are not difficult to select, but a blue and a red dye with a higher light fastness than 5 are not available. The dyes given in Table VI will be found of value for those purposes where acid dyes are required. Dyeing is commenced at 40°c, with 3% of sulphuric acid. The temperature is raised to the boil, and after dyeing for 15 min. a further 3% sulphuric acid is added. Dyeing is concluded 60 min. after the last addition of acid. It is desirable, after dyeing, to scour the Acrilan at 60°c. in a solution containing 2 parts of Lissapol NC and 1 part of soda ash per 1,000 parts of liquor. This scouring process removes some acid from the fibre and helps to improve the subsequent wet fastness. Unfortunately, the deleterious effect of dyeing with sulphuric acid on the handle of Acrilan cannot readily be reversed by scouring, as, at the temperature at which acid can be scoured out from Acrilan, dye also is removed.

Fig. 8 illustrates the effects of time and temperature upon the exhaustion into Acrilan of three level-dyeing acid dyes, and shows that dyeing at the commercial boil is essential. It appears from the results that complete exhaustion is then attained during the first 20 min., but dyeing must be continued further to allow the dye to diffuse from the surface layers of the fibre into the centre.

Acid milling dyes are much more difficult to apply to Acrilan than are the level-dyeing acid dves. It has been shown that high concentrations of sulphuric acid are still required in order to achieve good exhaustion of dye and good penetration of the dye into the fibre. Most acid milling dyes are applied from a dyebath containing 0.5-2.0% formic acid calculated on the weight of the goods. The temperature is raised to the boil, and then, gradually, up to 6% sulphuric acid is added. Dyeing is then continued in the usual way. With certain acid milling dyes it is advantageous to replace the formic acid by 1-2% ammonium sulphate. The aim in dyeing with this class of dye is to obtain some exhaustion into the surface layers of the fibre with formic acid or ammonium sulphate, and then, subsequently, to obtain, under more acidic conditions, good penetration of the fibre. The solubility of a short range of dyes and their fastness properties are detailed in Table VII.

Various means have been suggested for overcoming the danger of dye precipitation. One possible method is the use of solubilising agents ¹¹, and Dispersol VL is of value for this purpose. Another interesting possibility is the use of a cationic agent, which precipitates the acid dye, dyeing being carried out in the presence of a nonionic agent to keep the precipitate finely dispersed (personal communication from Mr. W. H. Hindle). Lissolamine A is suggested as a suitable cationic agent, with Dispersol VL as the non-ionic agent. Careful selection of acid dyes for use with this

TABLE VIII

Effect of Sulphuric Acid in Dyebath on Light

(Dyed in 40:1 liquor at 98° c. for 90 min.; soaped in 2/1,000 Lissapol NC + 1/1,000 soda ash at 60° c. for 20 min.)

for 20 mi	n.)	
Dye	H ₂ SO ₄ (168°Tw.) (%)	Light Fastness (Fade- Ometer*)
2% Coomassie Fast Yellow GS	1	2
(C.I. Acid Yellow 85)	3	3-4
	6	4-5
	10	4-5
2% Lissamine Fast Yellow 2GS	1	4-5
(C.I. Acid Yellow 17)	3	5-6
	6	5-6
	10	5
2% Croceine Scarlet 3BS	1	2
(C.I. Acid Red 73)	3	4
	6	4-5
	10	4-5
2% Solway Blue 2GS	1	4-5
(C.I. Acid Blue 40)	3	4-5
	6	4-5
	10	4-5
2% Coomassie Fast Grey 3GS	1	6
(C.I. Acid Black 48)	3	6
	6	6
	10	6

 A "cold" Fade-Ometer (i.e. one in which the patterns were in a humidified chamber behind a water-in-glass filter) was used, and tests have shown that generally on Acrilan the results do not differ by more than 0-5 grade from those obtained in daylight.

method is essential, as the dye-cationic agent complexes are individualistic in their behaviour.

Tests have shown that, with certain dyes, the amount of acid present in the dyebath can affect both the fastness to wet treatments and the fastness to light (Tables VIII and IX). At the present time no explanation is available either for the increased light fastness obtained with certain acid dyes applied in the presence of high concentrations of sulphuric acid, or their decreased fastness to low-temperature wet fastness tests. This last observation is contrary to expectation, although washing tests at high temperatures do show an improved fastness as the quantity of sulphuric acid in the dyebath is increased, resulting from greater penetration of the fibre by the dye. A severe alkaline scour after dyeing may eliminate the deleterious effect of acid on light fastness. This possibility has not been investigated, and, in any case, a scour more severe than that recommended removes appreciable quantities of dye from the fibre, which makes matching difficult.

When considering acid dyes for dyeing Acrilan, the difficulty of obtaining level results with mixtures of dyes, the danger of dye precipitation, and the effect of the acid on the handle of the fibre cannot be too strongly emphasised, although the importance of the handle of the fibre depends upon its ultimate use and the stage at which the material is dyed. For example, handle is of great importance to the yarn or piece dyer, but of much less importance to the loose-fibre dyer.

TABLE IX

astness of Acid Dyes	NC at 60°c. for 20 min.)	4 000
in Dyebath on the Wet F	(Dyed at 98°c. for 90 min.; "soaped" in 2/1,000 Limapol NC at 60°c. for 20 min.	
Effect of Sulphuric Acid	yed at 98°c. for 90 min.; "s	
-	9	

			(Dyed at b	18 C. IOF DU II	un.; soabed n	n Z/1,000 Lemmapo	4 NO at 500 C.	lor 20 min.)			
Dye		H,SO,	S.D.C. W	Jashing Test	No. 2		Once at 85°C.	• •		Washing Tos	t No. 4
(standard depth)			Colour	Acrilan	ning of Wool	Effect on Staining of Effect on Staining of Colour Acrilan Wool	Acrilan	ning of Wool	Effect on Staining of Colour Acrilan Wool	Acrilan	ng of Wool
Croceine Searlet 3B	* 0 *	63	2	4-5	*	60	*	63		3.4	69
			Į	9	•	ol	*	89		ī	67
Solway Blue 2G	:	69	4-5	10	10	8	4-5	*	1	4	3-4
		9	*	10	9	01	4	+	61	4	I
Solway Green G		8	10	10	10	69	10	10	1	10	*
(C.I. Acid Green 25)		9	*	10	- 2	89	10	9	99	10	10
		10	I	10	45	69	10	19	8	13	2
Coomassie Fast Grey 3G		60	*	45	10	60	10	- 9-9	1	10	*
		9	*	10	. 2	61	2	10	1	9	
		10	*	10	10	C4	10	9-4	CH	10	*

. ICI, The Postness Assessment of Textile Ducatuffs (1954).

TABLE X

Alkaline Milling Effect Staining of on Acrilan Wool Solubility and Fastness of Selected Chrome Dyes (Dyed with 3-8% sulphuric acid (168°Tw.) in 40:1 liquor at 98°c. for 90 min.; chromed in a bath containing 4% formic acid (85%) + 1.5-3-0% K₈Cr₈O₇ at 98°c. for 90 min.; "scaped" in 2/1,000 Lissapol NC + 1/1,000 soda ash at 60°c. for 20 min.) Colour 4-6 Effect Staining of on Acrilan Wool Effect Colour on S.D.C. Washing Test No. 2 Effect Staining of on Acrilan Wool Colour 1-5 Light (Fade-Ometer) 9 9 Insol. Insol. 2% 4% H₂SO, (168°Tw.) Sol. Insol. Solubility* Insol. 2% 4% HCOOH (82%) Sol. Insol. Sol. C.I. Mordant Yellow 10 C.I. Mordant Yellow 5 C.I. Mordant Orange 12 C.I. Mordant Red 7 Colour Index No. Solochromate Fast Orange 2R Dye (medium depth) Solochrome Yellow 2G Solochrome Flavine G Solochrome Red ER

Insol.

Insol. Sol.

> Insol. Sol.

C.I. Mordant Brown 38
C.I. Mordant Black 17
C.I. Mordant Black 9
C.I. Mordant Black 9

Solochromate Fast Bordeaux BN

Solochrome Brown RH

Solochrome Dark Blue B Solochrome Fast Navy 2R

Solochrome Black PV Solochrome Black PG

Bol. Sol. Sol.

Sol.

* Water of 5° hardness at 55°C., Bquor ratio 10:1.

• Water of 5° hardness at 55°c., † B = Borderline.

CHROME MORDANT DYES

Early experiments established that chrome dyes were applicable to Acrilan by an afterchrome technique but were not suitable for application by either a chrome-mordant or a chromate dyeing

As a class, the chrome dyes behave in a similar manner to acid dyes in the dyeing of Acrilan. Sulphuric acid, at a concentration of 4-8%, is essential to allow penetration of the fibre by the dye, and as a consequence care is necessary if dye precipitation is to be avoided. The chrome dyes vary enormously in their sensitivity to sulphuric acid: dves such as

icia. ayes such as	
Solochrome Yellow 2G	C.I. Mordant Yellow 10
Solochrome Flavine R	C.I. Mordant Yellow 8
Solochrome Brown RH	C.I. Mordant Brown 33
Solochrome Red B	C.I. Mordant Red 9
Solochrome Dark Blue B	C.I. Mordant Black 17
Solochrome Fast Navy 2R	C.I. Mordant Blue 9
Solway Blue Black B	C.I. Mordant Black 13
Solochrome Black PV	C.I. Mordant Black 9
Solochrome Black PG	C.I. Mordant Black 7

are relatively insensitive, and sulphuric acid can be used, with reasonable precautions, without fear of dye precipitation. Many chrome dyes are, however, extremely sensitive to sulphuric acid, and for this reason alone are not suitable for dyeing Acrilan. Table X lists the solubility of a number of chrome dyes with various acids in the dyebath. Use should be made of this type of information in dyeing, for with many chrome dyes, as with acid dyes, it is essential to commence dyeing with formic acid or ammonium sulphate and, at the commercial boil, when some absorption of dye has been achieved, to produce the final exhaustion and good penetration of the fibre filaments by slow addition of sulphuric acid to the dyebath.

In most cases it is not possible to chrome in the same bath, for two reasons—(i) chrome dyes are not completely exhausted on Acrilan, and therefore if the same bath is used for chroming, dirty dyeings will result; (ii) the high acidity of the dyebath would certainly cause difficulties in chroming, as serious destruction of most chrome dyes would result.

Chrome dyes are chromed on Acrilan from a bath containing formic acid and sodium dichromate. If sulphuric acid is used, serious destruction of dye results with most chrome dyes. The quantity of sodium dichromate used is also critical, as large amounts can, likewise, cause destructive oxidation of dye (cf. Fig. 9). On wool the possibility of oxidative destruction of chrome dyes is not great, presumably because of the presence of reducing groups in the fibre. On nylon, too, the effect is not great, because of the very limited capacity of dyed nylon for dichromate anions 14. Acrilan has no powerful reducing groups, and has a much greater capacity than nylon for dichromate anions, so that the liability of chrome dyes to be oxidised on this fibre can be understood. Generally, chroming should be carried out for 1 hr. in a bath containing 4% formic acid and 1--3% sodium dichromate. Medium depths are generally fully chromed with 1.5% sodium dichromate, but blacks require 3%.

Not all chrome dyes are suitable for application to Acrilan, owing to failure to realise their true colour. Examples of this behaviour are given by Kramrisch 5, and a further example is Solochrome Black WDFA (C.I. Mordant Black 11), which gives a dull violet when applied to Acrilan. The preferred chrome blacks for dyeing this fibre are Solochrome Blacks PG and PV. It has been suggested 15 that the addition of reducing agents, such as sodium sulphite, to the chroming bath will result in superior chromation of those dyes which prove difficult by the normal technique. Unfortunately, in many cases the reducing agents used reduce the dye as well as the sodium dichromate, and for this reason their use can lead to non-reproducible results.

It is considered that, for dyeing 100% Acrilan, chrome dyes are of value only for certain heavy dyeings, e.g. black, navy, dark brown, deep maroon, and bottle green applied to loose fibre or slubbing. The wet fastness properties of the results will be very high, but the light fastness will be generally inferior to that realised on wool. Table X lists the fastness of selected chrome dyes to the

most important agencies.

ACID LEVELLING METAL-COMPLEX DYES

Acid levelling metal-complex dyes may readily be applied to Acrilan by the identical method to that used on wool. A reduction in the quantity of sulphuric acid used to below 6% is not possible if good fibre penetration, resulting in good build-up and fastness properties, is desired. Table XI lists the fastness properties of Ultralan dyes applied to 100% Acrilan. Ultralan dves can be used to dve Acrilan, with good fastness, to blacks, navy blues, dark browns, maroons, and bottle greens. The absence of a blue dye possessing even moderate light fastness in pale and medium depths is a serious limitation to the use of this class of dyes for colouring Acrilan. Generally, the fastness to wet treatments of acid levelling dyes on Acrilan is high, but it is only on the borderline of suitability for the requirements of the slubbing dyer. With this class of dyes the necessity for using high concentrations of sulphuric acid constitutes a disadvantage in view of the effect upon the handle of the fibre.

NEUTRAL-DYEING METAL-COMPLEX DYES

This range of dyes for wool is not of particular value for dyeing 100% Acrilan except for very pale colours, where the high light fastness is attractive. The main difficulty associated with the use of the 2:1 metal-complex dyes is their poor penetration of the fibre filaments when applied from weakly acid or neutral dyebaths, so that they fail to realise on Acrilan the high wet fastness these dyes show on wool. The defect of poor fibre penetration is fully appreciated 2, and various methods have been suggested for overcoming the difficulty. The use of high quantities of sulphuric acid is not recommended, as many 2: I metal-complex dyes are demetallised under these conditions. Removal of the metallic atom is undesirable, as the unmetallised dyes possess lower wet and light fastness, and the

(Dyed with 8% an	(Dyed with 8% sulphuric acid (168°Tw.) in	Fastn 1 40 : 1 liquo	Fastness Properties of Ultralan D	of Ultralan 1	aped" in	2/1,000 Lissapo	1 NC + 1/1	,000 soda	Verilian 900 Lissapol NC $+ 1/1,000$ soda ash at 60° c. for 20 min.	r 20 min.)	
Dye	Colour Index	Light	S.D.C. Wa	S.D.C. Washing Test No. 2	No. 2	Pe	Perspiration I		IIV	Alkaline Milling	84
(standard depth)	No.	(daylight)	Effect on Colour	Staining of Acrilan	Joo M	Effect on Colour	Acrilan V	og of Wool	ĕ .	Staining	le of Wool
Ultralan Yellow R	C.I. Acid Yellow 99	9	4	10	20	*	5	4	100	10	10
Ultralan Orange G	C.I. Acid Orange 74	6-7	+	2	10	4-6	10	5	1-5	10	4-5
Ultralan Orange GR	C.I. Acid Orange 62	6-7	4 Yellower	10	2	4 Yellower	10	4-5	10	.02	4-5
Ultralan Orange R	C.I. Acid Orange 76	6-7	*	9	10	4-5	10	10	2-4	10	4-5
Ultralan Leather Brown 6R	1	9	*	10	22	4-6	10	10	4-5	10	10
Ultralan Red R	C.I. Acid Red 180	9	4-5	10	. 5	4-6	4-5	4-5	9-4	10	*
Ultralan Red B	C.I. Acid Red 212	9	3.4	10	55	4 Yellower	10	+	4-6	10	10
Ultralan Pink BN	C.I. Acid Red 186	10	*	9	20	*	4-5	*	*	10	4-5
Ultralan Bordeaux R	C.I. Acid Red 179	2-6	4-5	10	2	*	4-5	4-5	*	10	4-6
Ultralan Bordeaux B	C.I. Acid Red 187	5-6	*	10	10	4 Yellower	. 55	4-6	*	10	4-6
Ultralan Violet 5R	1	9-9	3.4	10	12	27	5	*	4 Redder	10	10
Ultralan Navy Blue B	1	4-5	4-5	10	10	10	10	4-5	9	10	2
Ultralan Blue 2G	C.I. Acid Blue 158	3.	4-5	10	2	10-1	10	4-6	9-4	10	10
Ultralan Dark Green B	C.I. Acid Green 35	2	4-5	10	10	4-5	10	10	10	NO	10
Ultralan Black RN	C.I. Acid Black 52	6-7	10	10	10	10	10	20	10	4	10

difficulty of obtaining reproducible results must also be considered. The effect of demetallisation upon light fastness is clearly shown in Table I. The use of carriers has been suggested as a means of utilising the 2:1 metal-complex dyes 15, but, although the laboratory results appear promising, the process does not yet appear to be in use commercially, and little is published concerning the effect of carriers on the mechanical properties of the fibre. In any event, as Acrilan is dyeable with many classes of dyes, it appears to be unremunerative to consider the use of carriers, which are usually troublesome, when other, more suitable dyes are available.

REACTIVE DYES

The Procion and Procion H types of reactive dyes may be applied to Acrilan from a bath containing 4% formic acid and 1 part of Dispersol VL per 1,000 parts of dye liquor. The wet fastness of these dyes applied to Acrilan is very high, but, with the dyes at present available, the light fastness is much lower on Acrilan than on cellulosic fibres. The high wet fastness results from a reaction between the dye and the basic groups in the fibre. Unfortunately, after the initial absorption of dye, practically no subsequent migration occurs, and difficulties in producing level results can be anticipated. Although, generally, it is considered that this class of dyes is of little value for the practical dyeing of 100% Acrilan, the use of Procion Brilliant Blue H7G is an exception if bright, fast, turquoise blues are required. This dye can, if desired, be shaded with Procion Brilliant Yellow 6G, but otherwise is not recommended other than in self-shades and for dyeing loose fibre or slubbing. If satisfactory dyeings with this dye are to be achieved, care should be taken not to apply it in pale colours, not to use more than 3% of Procion Brilliant Blue H7GS, and to give a scouring treatment after dyeing is completed.

BASIC DYES

Basic dyes can be applied to Acrilan by the same technique as that used to dye Orlon. Although the light fastness of basic dyes applied to Acrilan is much higher than that found with the same dyes applied to cellulosic fibres, the fastness is lower than on Orlon 42. For example, with ten dyes, including the new type of fast basic dyes, the light fastness was found, on average, to be 1.5 grades lower on Acrilan than on Orlon 42. Similar results have been obtained by Kramrisch ⁵. Although the wet fastness of basic dyes applied to Acrilan is high, it is not suggested that these dyes will be of value for Acrilan, except for the production of certain bright colours which are unobtainable with any other dyes, because of their unattractive dyeing properties in all except heavy depths. In certain cases, it may be desirable to use mixtures of basic dyes with disperse dyes in order to obtain a particular bright shade. It must be remembered, however, that disperse dye powders usually contain large amounts of colourless anionic dispersing agents. These agents can combine with basic dyes and produce colour spots. It is therefore suggested that, in addition to the control of pH

when applying basic dyes 1,5 and the use of a nonionic dispersing agent such as Dispersol VL or Lissapol N to assist the prevention of precipitation of a basic dye complex, Duranol and Dispersol printing pastes should be used, as these contain less anionic dispersing agent than the powder preparations.

DYEING TECHNIQUES AND CONCLUSION

No effort has been made in this paper to describe, in detail, practical dyeing methods. When dyeing Acrilan it must be remembered that the fibre is plastic when immersed in boiling dyebaths. For this reason care should be taken- (a) in cooling down before rinsing off; (b) when package-dyeing in a machine with a circulating pump, to avoid the use of high pressures; and (c) to avoid tensions in yarn dyeing or piece dyeing if the handle or the dimensions of the fibre are not to be seriously

Good results on 100% Acrilan have been obtained by— (a) piece-dyeing with disperse dyes on a winch or on a paddle machine; (b) yarn-dyeing with disperse dyes on a two-stick Hussong-type machine; (c) slubbing-dyeing either as tops in a central spindle holder or as packed hanks in a cage or can holder using a package-dyeing machine equipped with a circulating pump. It therefore appears that, provided that the right type of machinery is available, 100% Acrilan can be dyed in a wide variety of yarns with entirely adequate standards of fastness. It is wrong, however, to regard Acrilan as a fibre which, from the dyeing

viewpoint, behaves as does wool. Ultimately, less difficulty is caused if it is recognised that Acrilan is a new synthetic fibre, which must be considered in its own right as a dyeing problem. Generally, disperse dyes are the most attractive, although for specialised purposes azoic, acid, basic, chrome, metal-complex, and reactive dyes will be found of value.

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(MS. received 20th February 1958)

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Recent Developments in the Application of Azoic Dyes*

M. HÜCKEL

Meeting of the Manchester Section held at the Textile Institute, Manchester, on 18th October 1957, Mr. J. W. Reidy in the chair

The paper is concerned with recent knowledge of impregnation with Naphtol AS and of coupling conditions. Influence of the type of alkali in the impregnation bath on the dyeings and on the rate-of-absorption curves of various coupling components is discussed. The chemistry of the coupling of azoic combinations on vegetable fibres is outlined, and practical aspects of the buffering of the developing baths are considered. The base printing style also is briefly described.

Development of New Products

Since Naphtol AS was introduced there have been many new coupling components and diazo components placed on the market, and it is possible to produce today approx. 2000 different azoic dyes on the fibre. Some of these dyeings are not very satisfactory in certain details, but it is a remarkable fact that Naphtol AS, the parent of the coupling components, is still the most popular member of the range.

The tendency nowadays is to feel that all the simple coupling components and diazo components have been exhausted, and the newer products all seem to have larger molecules and are more complicated to produce commercially. However, new products are continually being produced to fill gaps in the colour range, as for example Fast Blue Green Salt B, which is the first product

yielding a green on Naphtol AS. Even in the apparently fully covered field of red and scarlet there are continual demands for dveings with additional fastness properties to meet new finishing techniques and consumer fastness demands, which means that this field, too, is continually expanding.

Impregnation

The principles of this stage of the process have changed relatively little over the years, and present fewer problems than the development process. The practical dyer knows that the coupling components of medium and high substantivity are more suitable for dyeing in long liquors, whilst Naphtol AS brands of low substantivity are primarily utilised on the pad. The experience of the past few years 5 has, however, shown that this classification is not rigid, as under suitable conditions.

The full text of this lecture has been published in the Canadian Textile Journal, 75, 57-65 (18th April 1958), and therefore an abridged version is published here.

even coupling components of low substantivity can be dyed in long liquor ratios, and Naphtol AS brands of high substantivity can be applied on the padding machine.

EFFECT OF TYPE OF ALKALI

Dealing firstly with the use of long liquor ratios, it is worth studying some experimental work on the effect on the impregnating bath of the type of alkali and its concentration.

Cotton yarn is impregnated in a bath (20:1 liquor ratio) containing per litre—

3 g. Naphtol AS dissolved in 3 c.c. Methylated spirit

1.5 c.e. Caustic soda (71° Tw.)

4.5 c.e. Water (40°c.)

3 c.e. Formaldehyde (33%) and additionally

30 g. Common salt

0, 2, 4, 6, 10 e.e. Caustie soda (71° Tw.)

When developed with a diazotised Fast Colour Base or Fast Colour Salt the depth of dyeing decreases with increasing concentration of caustic soda solution. The dyeing without additional caustic soda is brighter and even somewhat fuller than the other dyeings, almost regardless of whether the coupling was carried out at a high or a low pH value. A simple explanation of this phenomenon, which is limited to the less substantive Naphtols, cannot be given. It is possible that several factors act jointly in this case, e.g. effect on the particle size, affinity of the naphtholate in non-ionised and in ionised form, stripping action of the caustic soda solution, and other factors.

This is a very important observation, for if, in the case of Naphtols whose formaldehyde compounds have good solubility, the excess of caustic soda solution in the salt-containing bath is replaced by about 3 g. soda ash per litre, very deep dyeings are obtained. Hence it appears that, in the presence of formaldehyde, soda ash used as an alkali excess in the impregnating bath is more beneficial to the brilliance and the depth of the dyeings than are smaller quantities of caustic soda. It can furthermore be observed that the stability of the impregnated goods to atmospheric influences remains unaffected when 3 g. of soda ash per litre is used as an alkali excess, whereas it is lowered when smaller amounts of caustic soda solutions are used. However, with baths not containing formaldehyde, weaker dyeings are obtained with soda ash as an alkali excess than when caustic soda is used, at least with the different Naphtols which we have so far investigated. This behaviour obviously depends on the fact that the sodium salts of the free dye acids hydrolyse more easily than the sodium salts of the formaldehyde compounds, which are stronger acids 6

In the cases of Naphtols AS-RL, AS-VL, and AS-CA (C.I. Azoic Coupling Components 11, 46, and 41), which only form their formaldehyde compounds relatively slowly, the results depend on the experimental conditions, and a decisive factor is that the formaldehyde compound must have been formed completely. Naphtols AS-E and AS-SW (C.I. Azoic Coupling Components 10 and 7), as well as some highly substantive and easily

TABLE I

Naphtols whose Formaldehyde Compounds, with the addition of 3 g. of Soda Ash per litre and of Common Salt, give Full Dyeings

Naphtol	C.I. Azoic Coupling Component
AS	2
AS-D	18
AS-OL	20
AS-PH	14
AS-TR	. 8
AS-ITR	12
AS-BG	19
AS-BS	17
AS-RS	and the same of th
AS-LC	. 23

hydrolysable Naphtol AS brands, cannot be dyed satisfactorily in liquors containing soda ash. The same applies to those products which have to be dyed without formaldehyde, e.g. Naphtol AS-S (C.I. Azoic Coupling Component 32). A primary condition of this process is, however, careful control. In order to avoid precipitation, and to obtain adequate stability of the solution, the alcoholic naphtholate solution, prepared in the normal way by the cold-dissolving method, must be introduced slowly into the vigorously agitated soda ash bath at about 45°c.

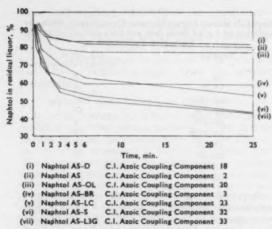
This working method, the "soda ash impregnating method", has meanwhile become very popular in practice, as it not only offers advantages when Naphtols of low substantivity are used in long liquor ratios, but it is also suitable for impregnations which are to be developed with diazo components sensitive to alkalis, such as Fast Navy Blue Salt RA and Fast Brown Salt VA.

In the dyeing of wound packages of regenerated cellulosic fibres, the utilisation of soda ash in the place of caustic soda solution as excess of alkali in the impregnation bath has a beneficial effect, as the swelling of the yarn is lowered, and the difficulties of penetration are avoided to a certain extent.

RATES OF ABSORPTION

Fig. 1 shows representative rate-of-absorption curves for equimolar solutions containing 0.005 m. Naphtol at a 20:1 liquor ratio at 30°c.7. Their most striking feature is the initial steep fall, although they tend to level off after 1-3 min. Thus with Naphtol AS-BR the initially steep curve becomes nearly horizontal after 6 min. This product not only has a high substantivity, but also goes on the fibre very quickly, so that its reputation for levelling very badly is easily understood. practical working the dyer can counteract the high affinity of Naphtol AS-BR by the use of auxiliaries with a retarding action, such as Remol AS or other polyoxyethylene products. Raising the impregnating temperature will not help at all in this case, as, contrary to the normal rule, Naphtol AS-BR has a higher affinity at higher temperatures.

In the case of Naphtols AS-D, AS-OL, and others of low substantivity the curves are moderately steep at the start, and become almost horizontal after 3 min. at the most. This means that the equilibrium between the Naphtol



F10. 1—Rates of Absorption of Naphtol AS Brands on Cotton from 0.005 M. Solution (liquor ratio 20:1)

on the fibre and the Naphtol in the bath is reached quickly. Such coupling components are very suitable for dyeing on the padding machine.

The curves of Naphtols AS-S, AS-LC, AS-L3G, and other highly substantive Naphtols, which at the beginning are steep and only gradually level off, show that these products, despite their higher substantivity, have a good levelling action, as their absorption is relatively slow and hence also uniform. On the pad the impregnation temperature is near 90°c. When working with long liquor ratios the impregnation is started at higher temperatures and, if required, the bath is gradually cooled to 30-40°c. In this case, however, it must be remembered that above 60°c, the Naphtol-formaldehyde compounds can be converted into methylene compounds, which are no longer capable of coupling. Therefore, in this process the formaldehyde addition to the naphtholate solution must be omitted.

In addition to this, the shape of the rate-ofabsorption curves permits some conclusions to be drawn for dyeing on the padding machine, which have been confirmed in practice. Fig. 1 shows that, apart from the extremely steep curve of Naphtol

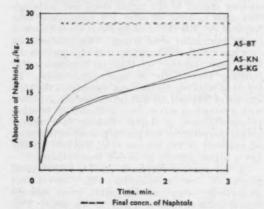


Fig. 2— Rate of Absorption of Naphtois A8-BT, A8-KN, and A8-KG by Cotton (liquor ratio 20:1, conen. 2 g./litre)

AS-BR, the reduction in concentration after 1 min. amounts to scarcely more than 25%. The dipping time on the pad is, however, only a few seconds. The decrease in concentration is therefore very low. We have investigated more closely the initial section of the curves of some highly substantive Naphtols, which were formerly dyed almost exclusively in long liquor ratios.

Fig 2 shows that the rates of absorption of Naphtols AS-KN and AS-KG are inferior to that of Naphtol AS-BT, despite an almost equal substantivity. The speeds of goods, thanks to progress in the technique of machine construction, are steadily increasing, and are at least 40 yd./min. in modern padding equipment, permitting an increase to 120 yd./min.

Developing

CHEMISTRY

Let us first consider the basic chemistry underlying the process of development. In the powder form the diazotised Fast Colour Base is a complex salt, which, when dissolved in water, decomposes into ions—

$$Ar\cdot N:NCl \Rightarrow [Ar\cdot N:N]^+ + Cl^-$$

Addition of a very weak base, e.g. silver oxide (Ag_2O) , leads in aqueous solutions to the diazonium hydroxide—

which also is a heteropolar ionic compound, of basic strength similar to that of ammonia.

Stronger bases, e.g. sodium hydroxide, however, initiate a process which, depending on the pH value of the solution, passes through a number of more or less stable diazo compounds, which vary amongst themselves in coupling capacity. The heteropolar diazonium hydroxide is first converted into the covalent diazo hydroxide—

longer basic, but is now acidic, since it may produce hydrogen ions, so that, with excess of sodium hydroxide, sodium diazoate Ar-N:N·ONa is formed.

The stage of the diazo hydroxide is of particular importance, as it is distinguished by the highest coupling potential. In this compound, however, the diazo nitrogen is least strongly bound, so that solutions of diazo hydroxides lose their coupling properties relatively quickly. Decomposition and self-coupling are assumed to occur—

$$Ar\cdot N:N\cdot OH \rightarrow Ar\cdot OH + N_1$$

 $Ar\cdot N:N\cdot OH + Ar\cdot OH \rightarrow Ar\cdot N:N\cdot Arylene\cdot OH$

The dyer should therefore prepare his developing baths in such a way that the equilibrium (i) lies as far as possible to the left, so that the amount of easily decomposable diazo hydroxide available at any moment is low, but is great enough for coupling to occur, according to the law of mass action.

COUPLING ENERGY AND COUPLING RATE

The different diazo components are coupled within distinct optimum pH ranges, and the diazo compounds of the Fast Colour Bases and the Fast Colour Salts may be classified into four groups (Table II). Within these groups there is a further

	TAB	LE II		
ning	of Di	970	Com	nonente

of Diazo Com	Diazo Components				
Coupling Energy	pH Range				
High	4-5				
Medium	5.5-6.5				
Low	6-7				
Very low	7-8-2				
	Coupling Energy High Medium Low				

subdivision into "rapid", "medium", and "slow" according to the coupling rate.

The terms coupling energy and coupling rate refer to the maximum reactivity of the diazo component and the overall rate of coupling of the two components in the optimum pH range.

The coupling energy of the diazo compounds is determined by the nature and the position of the substituents in the molecule. The greater the number of negative substituents in the molecule, and the stronger their electronegative effect, the

TABLE III Substituents of Diazo Components in order of

	Influence on Coupling Energy
	NEGATIVE SUBSTITUENTS (decreasing electronegative effect)
Group 1	$NO_3 > CI > Br > CF_3 > CN$
Group 2	80 ₃ ·Alk > 80 ₃ ·Ar > 80 ₃ ·NAlk ₄ > 80 ₃ ·NH·Ar
Group 3	CO·Alk > CO·Ar > COOAlk > COOAr
	Donners Cringmanicus

(increasing positive effect) $\mathrm{CH_3} < \mathrm{C_9H_5} < \mathrm{CH_9 \cdot C_9H_5}$ Group 4 Group 5 O-CH₈ < O-C₂H₈ Group 6 $O{\cdot}C_{\mathfrak o}H_{\mathfrak o}$ Group 7 NH-Alk

NH-C.H.

more the equilibrium (i) lies to the right-hand side, and the more vigorous is the coupling. In other words, the negatively substituted diazo

Fast Colour Bases* and Fast Colour Salts

Group 8

	GROUP I— HIGH COUPLI (pH 4-5)	ING ENERGY	GROUP II—MEDIUM COUPLING (pH 5.5-6.5)	ENERGY
	(px1 + 0)	C.I. Azoie Diazo		I. Azoic Diazo
		Component	Banid Counting Bata	Component
	Rapid Coupling	Rate	Rapid Coupling Rate Fast Scarlet TR Base†	46
Fast (Orange R Base*	7	Fast Bordeaux Salt OL	30
	Orange GR Base	6		30
	Scarlet GGS Base	3	Medium Coupling Rate	-00
	Red GG Base	37	Fast Red FG Base	26
	Red 3GL Base special	9	Fast Red KB Base	32
		and the state of t	Fast Red FR Base Fast Red TR Base	11
	Medium Coupling	Rate	Fast Red IR Base	29
Fast Y	Yellow GC Base	44	Fast Red RC Base	10
Fast (Drange GC Base	2	Fast Corinth Salt V	39
Fast C	Orange Salt LG	18		The second second
Fast (Orange Salt GGD	16	Slow Coupling Rate	91
Fast 8	Scarlet G Base	12	Fast Scarlet Salt LG	31 25
Fast 8	Scarlet RC Base	13	Fast Red SW Base special† Fast Red ITR Base	42
Fast I	Red RL Base	34	Fast Red KL Base special	4.6
Fast l	Red GL Base	8	Past Ned KL Dase special	
Fast 1	Red Salt AL	36	GROUP III LOW COUPLING E	NERGY
Fast 1	Brown Salt RR	_	(pH 6-7)	
Fast 1	Bordeaux Salt BRH		Rapid Coupling Rate	
Fast (Garnet Salt RH	_	Fast Corinth LB Base	43
			Medium Coupling Rate	halos Milball
	Slow Coupling 1	Rate	Fast Violet B Base	41
Fast	Golden Orange Salt GR	19	Fast Violet F Base	
Fast (Orange Salt RD (A)	49	Fast Blue RR Base	24
Fast 8	Scarlet Salt AFA	50	Fast Blue BB Base	20
Fast 8	Scarlet Salt VD	17	Fast Black Salt G	45
Fast 1	Red B Base	5	Fast Black Salt ANS	-
Fast 1	Bordeaux GP Base	1	Slow Coupling Rate	107-10.3001
Fast 1	Bordeaux Salt BD	40	Fast Red RBE Base†	15
Fast (Garnet GC Base .	27	Fast Blue Green Salt B	-
Fast (Garnet GBC Base	4	GROUP IV- VERY LOW COUPLIN	G ENERGY
Fast (Olive Salt BR	2000	(pH 7-8·2)	
Fast 1	Brown Salt V (A)	21	Medium Coupling Rate	
	Navy Blue Salt RA	ALL SALES TO SERVICE TO	Variamine Blue Salt B (N)	35
	Black Salt K	38	Variamine Blue Salt RT (N)	22
	Black Salt BTL	-	Fast Black Salt B	-
			Slow Coupling Rate	
All the F	ast Bases, other than those	indicated by a dagger (†).	Variamine Blue Salt FGC	47
have corre	ast Bases, other than those esponding Fast Salts, e.g. Fas	t Orange Sait B, of similar	Fast Blue B Base	48
coupling p	n Oper sien.		A THE STREET AT MARKET	200

compounds must be coupled at relatively low pH values. Positive substituents, however, shift the equilibrium (i) to the left, for they lower the coupling energy and prolong the time of coupling. It is therefore advisable in these cases to develop at relatively high pH values.

Table III shows the different substituents of the diazo compounds, classified according to the strength of their influence on the coupling energy.

Table IV contains all Fast Colour Bases and those Fast Colour Salts which have no corresponding Fast Bases, subdivided according to their coupling energies and coupling rates into the above Groups I–IV (Table II). Table IV includes diazo components which have been put on the market recently.

COUPLING COMPONENTS

As a rule, the decisive factor determining the course of the coupling is the diazo component.

TABLE V Coupling Components

 $\begin{array}{ccccc} & pH \ 6\cdot2-7\cdot0 \\ Naphtols & AS-BT & AS-KN & AS-KG \\ & pH \ 5\cdot5-6\cdot5 \\ Naphtols & AS-LB \cdot AS-SG & AS-SR \end{array}$

Thus the Fast Colour Salts and the diazotised Fast Colour Bases may differ from each other in their coupling energies, depending on their chemical constitutions. The majority of coupling components, however, react rapidly, so that in general the course of the reaction is not influenced by the Naphtol. Only a few Naphtol AS brands couple so slowly that they become the components determining the course of coupling. These are arranged according to increasing coupling energy in Table V. The pH ranges given can serve only as guides, since they depend also to a large extent on the coupling energies of the Fast Colour Salts or diazotised Fast Colour Bases which are employed in each case.

BUFFERS

One of the most important developments has been in the study of buffers, and the realisation that there is no universal buffer, but that the buffer must be selected to meet the specific requirements of the diazo components.

Tables VI and VII show the pH values obtained with the buffers most commonly used in Naphtol AS dyeing practice.

PH of Acetic Acid-Sodium Acetate Buffers
(1.2 ml. Acetic acid (50%) per litre)

Sodium Acetate cryst. (g./litre)	pH
_	3-35
2.5	4.90
5.0	5.25
10-0	5.55
20.0	5-80

These two tables reveal the following characteristic features of the two buffer mixtures. While mixtures of acetic acid and sodium acetate

TABLE VII

pH of Monosodium Phosphate-Disodium Phosphate Buffers

(4 g. Disodium	phosphate	per liti
Monosodium		pH
Phosphate (g./litre)		
9.2		6.2
4.8		6.4
2.4		6.6
1.2		6-8
0.6		7-0

(The concentrations in g./litre correspond approximately to a liquor ratio of 20:1)

in the concentrations normally used in practice always give pH values lying between 5-0 and 5-5, the practical phosphate buffer mixtures cover the range 6-2-7-0. The two buffer mixtures are therefore preferably employed in developing liquors which contain either diazo components of medium coupling energy (Group II, acetate buffer) or those of low coupling energy (Group III, phosphate buffer).

In practical working it is not necessary to adjust too exactly to the theoretically optimum pH range. In general the rough differentiation by Groups I–IV is sufficient. Thus the adjustment of the pH of diazo solutions by the addition of buffers is much simpler than may appear at first glance.

COUPLING CONDITIONS

The main coupling conditions for the various groups of diazo components may now be considered

Group I

In view of the high coupling energy, no buffer addition is necessary, but an alkali-binding agent is required to compensate for the caustic alkali brought over by the impregnated material.

Fast Navy Blue Salt RA and Fast Brown Salt V or VA require special attention. Both these Colour Salts, which are aminoazo compounds containing strongly electronegative substituents, are distinguished by their high coupling energy, and have in addition a deep brown colour and a certain substantivity for cellulose. In order to prevent more Colour Salt from being adsorbed on the fibre than is required for coupling the Naphtol fixed on the fibre, which would dull the colour and impair the fastness, a small deficiency of diazo compound is maintained in the presence of a high concentration of acetic acid. The high coupling energy of these two products justifies the addition of an amount of acetic acid which is larger than is normally required for this group. The acid acts in a similar manner to a retarding auxiliary for dyes with pronounced substantivity. This method of working has become very popular in practice under the name base-deficiency process 9.

A modified form of this process is useful for the application of some of the newer products which have recently been put on the market, and which are no longer simple benzene derivatives, but compounds having large molecules with similar

characteristics to those of Fast Brown Salt V and Fast Navy Blue Salt RA. These new products are—

Fast Olive Salt BR Fast Bordeaux Salt BRH Fast Garnet Salt RH Fast Black Salt BTL,

Group II

Addition of a buffer is required with long liquors (acetic acid-sodium acetate is normally suitable).

In the case of Fast Colour Salts containing aluminium sulphate, it is very difficult to influence the pH by making part of the aluminium sulphate basic. Even if more than half of the aluminium sulphate has become basic, the pH will rise only a little above 4. When all the aluminium ions have been precipitated as hydroxide, the pH rises suddenly to 7. Great care is therefore needed with these Fast Colour Salts, and it is better to use diazotised Fast Colour Bases or Fast Colour Salts containing no aluminium sulphate (e.g. special brands such as Fast Red Salt TRA and Fast Orange Salt RDA), which can be buffered with acetic acid and sodium acetate. In addition, they offer the advantage of not causing any deposits in the dyeing of wound packages.

Group III

Addition of a buffer is essential to long liquors of these slowly coupling diazo components (a phosphate buffer is suitable).

As these products are best coupled in nearly neutral solutions, they frequently used to be applied by completely neutralising the excess of mineral acid in the diazo solution with sodium bicarbonate, and developing without further addition of alkali-binding agents. In such cases the pH rises above 7, which results in destruction of part of the diazo compound, which can hardly be controlled. The diazo compound of Fast Red TR Base is one of the rare substances whose solution remains relatively stable even in the alkaline region. As a number of diazotised Fast Colour Bases do not couple at all in the alkaline region, the utmost care must be observed when working by this method.

In recent years the use of phosphate buffers has become very popular. Thus the solution of the diazotised Fast Colour Base or of the Fast Colour Salt, which, particularly in the dyeing of wound packages, should not contain aluminium sulphate, may first be completely neutralised with sodium bicarbonate, and then the phosphate buffer (Table VII) is added. The phosphate concentrations will depend on the liquor ratio, and for a ratio of 20:1, for example, they will be about—

2 g. Disodium phosphate per litre 4 g. Monosodium phosphate per litre.

Double these concentrations will be used for a liquor ratio of 10:1, and four times for 5:1.

Solutions of Fast Colour Salts which are stabilised as zinc double salts, however, cannot be buffered with orthophosphates, as insoluble precipitates of

zinc phosphate would form, causing difficulties, particularly in the dyeing of wound packages.

For development with diazotised Fast Colour Bases, there is a smooth and safe method by which the acidity of the mineral-acid diazo solution is reduced, not by the addition of sodium bicarbonate, but directly with a phosphate buffer, adjusting at the same time the desired pH value. The excess of hydrochloric acid is removed by the reaction—

 $Na_{2}HPO_{4},12H_{4}O + HCI = NaH_{2}PO_{4},2H_{2}O + NaCl + 10H_{2}O$

The quantity of hydrochloric acid that must be neutralised after diazotisation can be seen from the tables (generally applicable to 1 kg. of base) supplied by the dye manufacturers.

Further, with long liquors and prolonged dyeing times, the material impregnated with alkaline naphtholate solution carries caustic soda into the developing solution, when the following reaction

 $NaH_2PO_4, 2H_2O + NaOH + 9H_2O = Na_2HPO_4, 12H_2O$

The amount of caustic soda fixed per kilogram of material, and the amount introduced into the developing bath, can also be seen from the tables and graphs of the dye manufacturers.

The correct adjustment of the phosphate buffer is easily checked with indicator paper.

For cases where a phosphate buffer cannot be used, e.g. with Fast Colour Salts stabilised as zinc double salts, or where phosphate buffering is inadequate, since both coupling and diazo components have too low coupling energies, we have worked out the following very interesting method.

We had found 10 that chromic salts of weak organic acids, e.g. normal chromic acetate, form complex compounds with the caustic soda carried over from the impregnating bath, the pH values of which are close to neutrality for equivalent amounts of caustic soda and chromic salt. About 8 parts by weight of normal chromic acetate are required for 1 part by weight of solid sodium hydroxide. In the case of Fast Colour Bases, the diazotised base is almost neutralised with sodium bicarbonate, as described previously, and normal chromic acetate is introduced into the developing bath in quantities equivalent to the amount of caustic soda solution. Chromic acetate can be used as the alkali-binding agent only with solutions of those Fast Colour Salts which contain no aluminium sulphate, or only a negligible quantity. This working method is of distinct advantage when Fast Red Salt KL is applied to packages.

Group IV

These products have only very low coupling energy, but buffering as for Groups II and III is not usual, for these Fast Colour Bases and Fast Colour Salts are generally considered unsuitable for application from long liquors for long times of dyeing.

It is important to note that, whereas the Naphtol dyer, working with long liquors for relatively long dyeing times in pack-dyeing equipment, becks, jigs, etc., must watch the buffering capacity of his developing liquors very carefully, conditions are

generally much simpler with continuous development on the padding machine.

Batchwise development on the padding machine occupies an intermediate position. In this process the material may be piled or batched after being padded with the developing liquor, which in certain cases is buffered to attain complete coupling during piling or batching. In the majority of cases, however, the dyer operates continuously in such a manner that the material padded with the developing liquor has first an air passage of about 30 sec., and immediately afterwards enters the open-width washing machine with as many boxes as possible. By this method it is possible to complete the coupling, even of slowly coupling components, by adding to the first box or to the first two boxes of the open-width washing machine hot water, a lukewarm solution of sodium carbonate, or a warm solution of sodium bicarbonate.

An excess of acetic acid is often used in the developing solution, partly to ensure that a sufficient quantity of alkali-binding agent is present, which should be checked at regular intervals, and also to retard coupling and thus obtain better penetration of the goods with the developing solution. Coupling begins slowly during the air passage, and is completed during the passage through the subsequent liquors.

The practical dyer normally prefers a passage through a hot water bath at 80-90°c. with a constant overflow. This method of working has the advantages that there is always a high temperature promoting coupling, and at the same time the excess of diazo compound and its decomposition products are removed by rinsing, and that the process is easily controlled. Only in difficult cases is it necessary to use a lukewarm bath containing about 10 g. soda ash per litre, or, according to our latest experiments, a warm bath containing about 10 g. sodium bicarbonate per litre. In these two methods frequent checking of the baths is, however, necessary to ensure that sufficient amounts of the requisite chemicals are always present.

Completion of coupling by one of these methods is essential with Fast Colour Salts and diazotised Fast Colour Bases of Groups III and IV, as well as with coupling components of the type of Naphtol AS-BT, which also have a decisive effect on the coupling conditions. Further details are given in the relevant leaflets of the dye manufacturers.

PRINTING

In the printing of thickened diazo compounds on goods impregnated with coupling componentthe base printing style—the coupling energy of the components, as with continuous dyeing on the padding machine, plays a less important rôle than is the case in dyeing from long liquors. Printing is normally carried out with an excess of 10-40 ml. of acetic acid (50%) per kilogram of printing paste. The acetic acid evaporates during the drying of the prints in the drying chamber or on the drying cylinder, and consequently the prints must pass through the pH value offering optimum conditions for coupling. Buffering or special pH control is not, in general, necessary. The excess of acetic acid offers furthermore the advantage that the dye is formed gradually in the interior of the material, and not only on its surface. Good penetration is obtained in this way, and the formation of decomposition products on the surface, which would dull the colour, is avoided.

An addition of about 100 g. urea per kilogram of printing paste has proved to be very beneficial during the dissolution of certain Fast Colour Salts,

> Fast Brown Salt V Fast Black Salt ANS Fast Garnet Salt RH Fast Bordeaux Salt BRH.

The solubility and the stability of the products are thereby increased, and the resulting prints are fuller and more level.

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(MS. received 15th January 1958)

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Discussion

Professor R. H. Peters: Can the author define more specifically what is meant by "coupling ? How are the rates of coupling determined? Finally, how far are the coupling rates associated with the classification-fast, medium, and slow?

Mr. HÜCKEL: According to the literature relating to the application of Naphtol AS "coupling energy" is the maximum reactivity of the diazo component with the coupling component or vice versa which can be achieved in a specific pH range. It has nothing to do with the energy which is released or consumed during a chemical reaction. For practical purposes the coupling energy represents a measure of how far the pH of the developing bath can be reduced without loss in the depth of the resulting dyeing.

The rate of coupling can be determined by comparing known and unknown diazo components during the developing process. It is not possible to define it numerically, as it varies according to the dyeing method employed (pad, jig, or packagedyeing machine). Its practical importance is that it enables diazo components of equal coupling energy and equal rate of coupling to be chosen for mixtures, thus avoiding unsatisfactory dyeings.

Mr. J. Greenwood: In the dyemakers' instructions for azoic dyeing, most of the bases in Groups I and II are recommended to be used in 50° excess. Is this excess necessary in view of the pH controls which the author mentions?

Mr. HÜCKEL: The reaction between coupling and diazo components proceeds according to the law of mass action. A greater or less excess of the diazo components must be added, depending on the pH and the temperature, in order to shift the equilibrium as far as possible in favour of the resulting azo dye.

When dyeing in long liquors the excess should therefore, even under the most favourable conditions (developing near the neutral point), be not less than 50%. Exceptions are only those Fast Colour Salts of very high coupling energy, e.g. Fast Navy Blue Salt RA, which are coupled according to the "base-deficiency process" mentioned in the lecture.

When dyeing on the pad or when using the "base-printing method" the procedure is much simpler. Normally no excess of diazo component is used, as in the case of Fast Navy Blue Salt RA.

Mr. S. W. Leech: Has Herr Hückel any experience of decrease in rubbing fastness due to subsequent application of stain-resisting finishes?

Mr. HÜCKEL: Our experience has shown that wax, fat, and fatty derivatives in most cases impair the fastness to rubbing of azoic dyeings. Careful aftertreatment of the dyeing is extremely

important, as carefully aftertreated dyeings show a much smaller decrease in fastness to rubbing than those with impaired rubbing fastness previous to finishing.

Mr. H. R. Hadfield: I can confirm that azoic combinations possess high wet fastness when applied to synthetic fibres, provided that wet treatment is carried out below 100°c. Unfortunately, many synthetic fibres are given a permanent pleating process above 100°c. using steam. Under these conditions a perfectly applied dyeing can "blind", i.e. pigment can migrate to the surface and show a change of colour and poor rubbing fastness. This behaviour represents a very serious limitation in the use of azoic dyes for synthetic fibres. Fortunately, azoic blacks do not show this behaviour.

Mr. HÜCKEL: I agree that under extreme conditions and with unsuitable azoic combinations, even when the dyeing is perfectly applied, the results can show a loss in rubbing fastness after the permanent pleating process. By "extreme" I mean conditions which exceed the normal treatment at 120°c. for 10 min. using steam. We also were able to achieve good results with azoic blacks, and recently with azoic navy blues, under normal conditions.

COMMUNICATION

Adsorption at Organic Surfaces*

III—Some Observations on the Constitution of Chitin and on its Adsorption of Inorganic and Organic Acids from Aqueous Solution

C. H. GILES, A. S. A. HASSAN, (Miss) MARGARET LAIDLAW, and R. V. R. SUBRAMANIAN

Adsorption of mineral and organic acids, from aqueous solution, by chitin prepared from the carapace of Nephrops norvegicus has been studied.

Full elementary analyses suggest that chitin does not consist entirely of poly-N-acetylglucoamine as usually supposed, but that about one-eighth of the amino groups are unacetylated. The material appears to be highly crystalline.

Mineral acid anions are adsorbed at all the acetamido or amino groups of the substrate at pH about $2\cdot5$. At pH $< 2\cdot5$ very greatly increased adsorption occurs, much beyond stoichiometric quantities. Organic acids, both aliphatic and aromatic, are much less strongly adsorbed than mineral acids, and < 10% of the total amount of the substrate appears to be accessible to them. Two opposing factors appear to determine the amount of an organic acid adsorbed at moderate acidities: (a) the crystallinity of the substrate, and (b) the length of the conjugate chain of the anion.

The main source of anion affinity under acid conditions appears to be physical attraction between the anion and the water-solvated glucosidic residues of the chitin molecular chains. Hydrogen-bond affinity appears to be weaker than the ion-ion affinity or that due to the van der Waals attraction of large aromatic anions, and does not appear to influence the extent of adsorption under acid conditions.

Introduction

Chitin is one of the most important of natural structural substances. In the animal kingdom it occurs as a principal constituent of the protective cuticles of insects and crustacea, and in the vegetable kingdom it forms part of the cell walls of some fungi and micro-organisms.

Chitin is a tough, highly crystalline substance. Chemically it is regarded as a polymer of N-acetylglucosamine with 1:4-linkages 5-10, i.e. as a derivative of cellulose (I) in which one hydroxy group in each glucose unit is replaced by the acetamido group (II)—

[†] For an account of the biological importance of chitin see Richards ². Kneoht and Hibbert ² give a useful survey of its general properties and occurrence, and review some early work on its constitution.

II- Chitin (principal repeating unit)

though, as will be discussed below, this formula may require slight modification. Chitin is relatively stable towards acids, alkalis, and oxidising agents. In its natural state it occurs in intimate association with many other substances, including protein, and in crustacea with calcium carbonate and colouring matters, and its separation usually requires rather lengthy treatments with aqueous solutions of acid and alkali.

The adsorption properties of chitin have been almost entirely neglected. Knecht and Hibbert ⁴ described some qualitative tests of its dyeing properties, and while the present work was in progress Hackman ¹¹ reported some quantitative experiments upon its adsorption behaviour with aqueous solutions of proteins.

Containing as it does chains of glucosidic units and a high proportion of amide groups, chitin is chemically similar both to cellulose and to nylon and the protein fibres, and it was therefore thought that a study of its adsorption properties might reveal interesting similarities to and dissimilarities from these groups of fibres. The present and the two succeeding papers are therefore devoted to a description of the results of a broad survey of the adsorption behaviour of chitin towards inorganic and organic acids and some sulphonated azo dyes, and to a comparison of its adsorption properties with those of cellulose.

Experimental

RAW MATERIAL

The starting materials used in this work were the shells of the Norwegian lobster (Nephrops norvegicus), the common crab, the edible lobster, shrimps, and prawns; and also the lining of the shell of the common lobster. The last-mentioned. which forms a thin colourless transparent film, containing little or no calcium carbonate and over 50% chitin, would be the preferred material, were it not difficult to obtain in sufficient quantity. Nephrops shells were the next in order of preference, because they are more readily comminuted; they were therefore used for all the quantitative tests in the present work. Some of the tests were made on finely powdered material. All the types of shell used are tough and hard in texture, and to reduce them to a fine powder, even after the preliminary chemical treatments, prolonged mechanical grinding is necessary; the shell fragments are so hard in fact that in this process some material from the balls used in milling is introduced into the powder (cf. Table II). In order to ensure that this impurity did not interfere with the present tests, silica balls were used, since silica does not readily adsorb anionic compounds from aqueous solution. Reference to differences in adsorptive

properties between ground and unground material is made below.

TABLE I
Effect of Pepsin Hydrolysis on Chitin-containing

	Raw N	laterial	
Source	Incubation Period (37°C.)	Nitrogen (%)	Biuret Reaction
Lobster shell	Nil	7-3	+ (strongly)
lining	1 hr.	7.5	+
	2 hr.	6.9	-
	3 hr.	6.8	-
	3 days	6.6	-
	4 days	6.8	-
Nephrops-	6 days	7-3	+ (alightly)
Broken	7 days	7-2	+ (slightly)
shells	8 days	6-6	+ (slightly)
Finely ground	8 days	6.9*	-

* Ash-free figure; ash content (silica) 6-3%.

Three methods of purification have been examined, viz. Clark and Smith's process 12, Thor's process ¹³, and a new process using enzyme hydrolysis. Clark and Smith removed calcium carbonate with cold dilute nitric acid, and protein by hydrolysis with boiling 20% aqueous sodium hydroxide for 4 hr. Thor's method is milder, sodium carbonate solution being used as the hydrolysing agent. Enzymatic treatment was examined as a possible method of removing protein without causing hydrolysis of the acetamido group. since the attack should be specific for the peptide linkages in the protein molecule. The protein in crustacean shells has a high proportion of tyrosine (Fraenkel and Rudall ^{8, 9}) and thus should be hydrolysed by treatment with pepsin. Examination of the product by the biuret test showed that this procedure did not completely remove protein from broken shell fragments, probably owing to incomplete penetration by the enzyme. The experiment was therefore repeated using finely ground (ball-milled) starting material, and the protein then appeared to be removed completely. Details are given in Table I. Unfortunately, the product contained not only silica, but some natural colouring matter also, which could not be fully extracted, though it could be destroyed by bleaching with potassium permanganate solution followed by sodium bisulphite.

The analytical data (Table II), however, do not show that enzymatic treatment of shells offers any substantial advantage over the shorter process of Thor, which was therefore used for preparing most of the chitin used in this research.

SEPARATION OF CHITIN

The shells, broken to about \$\frac{1}{4}\$ in., were successively treated with—\$(a)\$ cold 5% aqueous hydrochloric acid for 10–20 hr., (b) water, (c) boiling 1% aqueous sodium carbonate containing 0.02% of an anionic detergent, oleyl sodium sulphate (Lissapol C, ICI) (Thor used soap) for 8 hr., (d) water, (e) cold 5% aqueous hydrochloric acid for 1–2 hr., (f) water, and (g) sodium bicarbonate solution (treatment as before); finally the product was well

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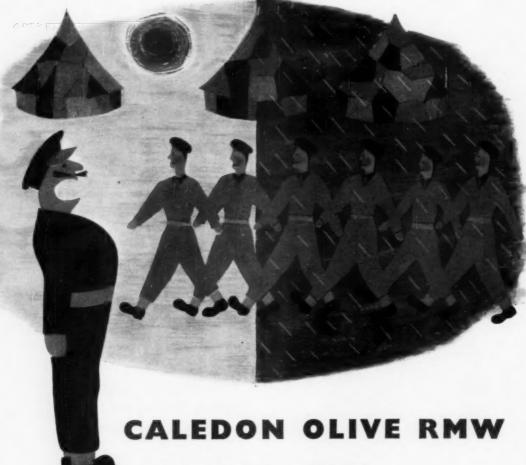
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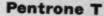
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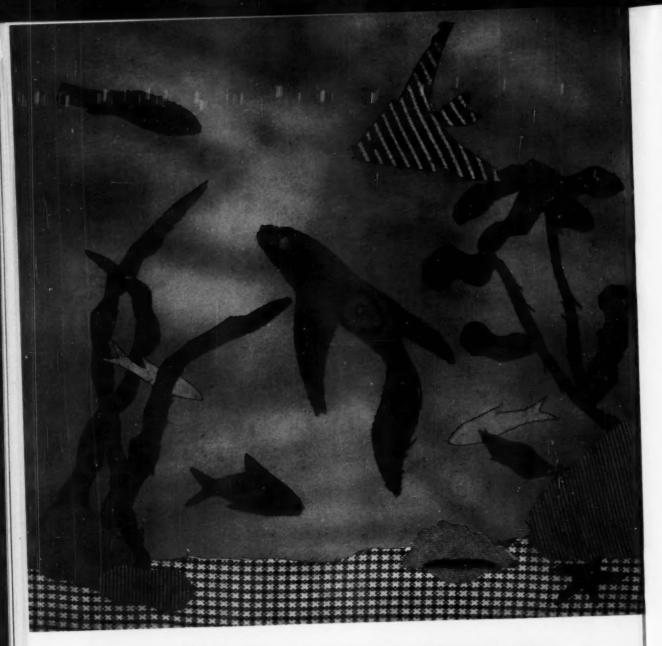
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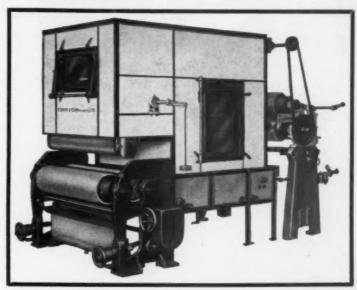
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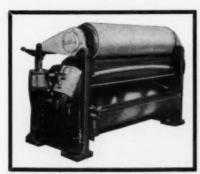


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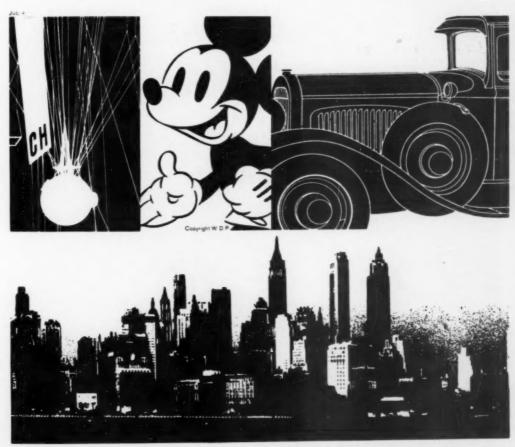
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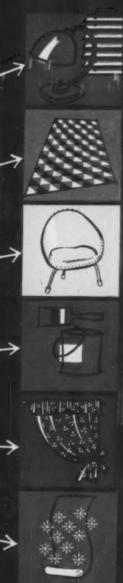
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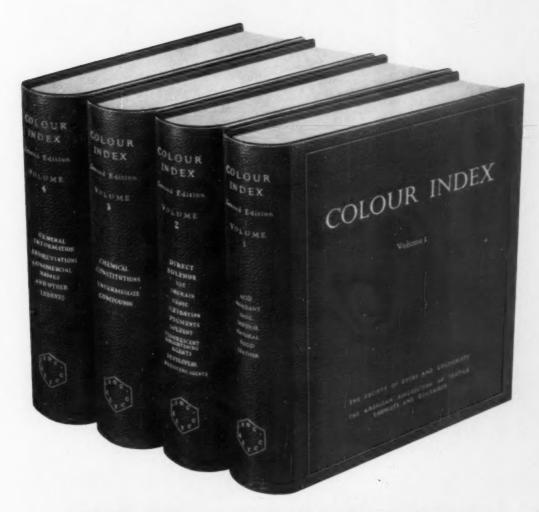
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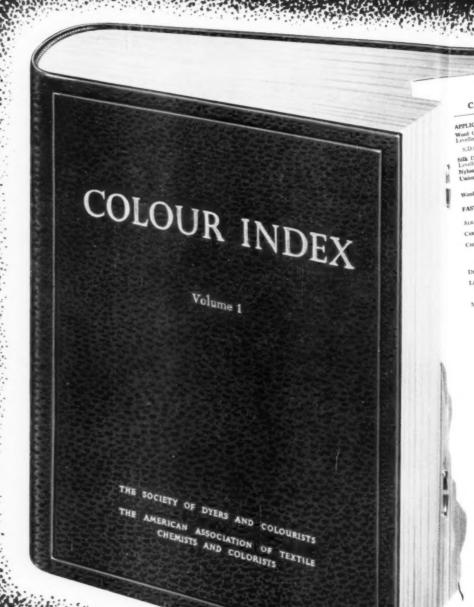
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APPLICATION	from a sulphuric acid dyebath				YDC
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Levening To	est Migration 3		Aigen Tartras	TF	CCC
S.D.C. Migration	good St { Divering method 1 } Migration 3 } nic or sulphuric acid dyebath good ormic acid dyebath dyebath sylon dyed, silk slightly stained, or PRINTING		Amacid Yellor	MCG, RX, XX	KKK
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A - (American) The methods of The American Association of Textile Chemists and Colorists

B— (British) The methods of The Society of Dyers and Colourists

C — (Continental) The methods of The German Fastness Committee

An important feature of Part I is the sub-division of each usage group into hue groups — Yellow, Orange, Red, Violet, Blue, Green, Brown and Black. The hue classification has been made by reference to a Hue Indication Chart which has been designed specially for the purpose. A copy of the chart is included with each set of volumes.

PART II is one volume of over 800 pages. It contains data relating to chemical constitution, method of preparation, patent and literature references relating to manufacture or constitution, solubilities and reactions in substance for approximately 3,800 dyes and pigments. The 1st Edition recorded 1,316 constitutions, and this very big increase is partly due to the fact that many co-operating dye manufacturers have, for the first time, disclosed the constitution of certain of their dyes. In addition there is the information contained in the reports compiled by Allied Investigating Commissions on the German Dye Industry.

The dyes are arranged in order based on their chemical constitution following broadly the lines of the 1st Edition. A number of refinements have been introduced and there are two new sections, viz. Azoic and Phthalocyanine.

Each entry carries a five digit reference number. These numbers do not progress in unit steps as provision has been made to allow the insertion of new constitutions in later editions or supplements without having to change the numbers now allocated in the 2nd Edition. Each entry also carries the complementary Part I designation thus providing easy cross reference to the data in Part I. It also contains alphabetical and empirical formula indexes to all the Intermediates relating to individual dye entities.

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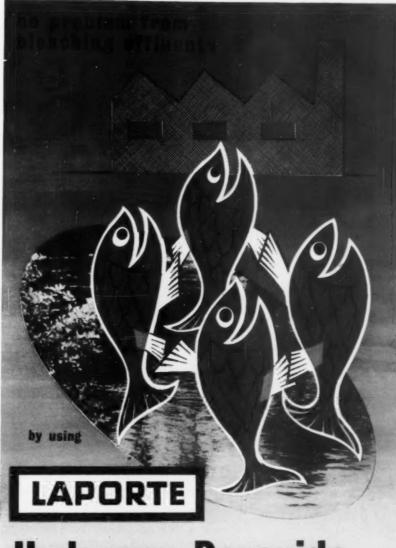
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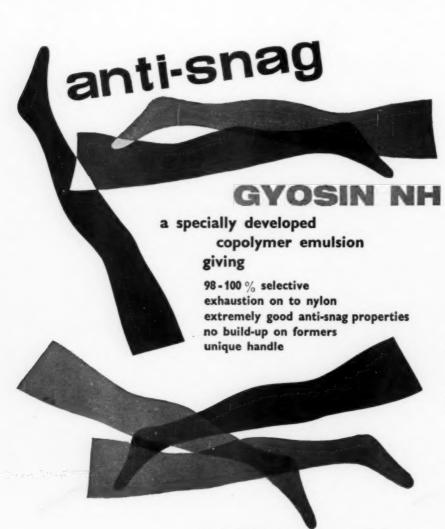
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TABLE II
Elementary Analytical Data on Chitin Samples

	Lieu	ientary Analytical Data on Ch	itin Sampi	CS		
	Source	Treatment	DILIDED AT	Analysi	s (%)	
			C	Н	N	CO-CH _a
(a)	Broken shells of common lobster*	Clark and Smith process	44.2	7.0	6.9	-
(b)	Lining of shells of common lobster*	Clark and Smith process	44.0	(5-6 (?))	6.7	
(e)	Broken shells of Nephrops norvegicus*	Thor process	44-2	6-4	6-5	-
(d)	As (c)*	Thor process†	44-6	6-3	6.7	-
(e)	As (c), ball-milled	Thor process	45-32	6-71	7-11	
(f)	As (c), not milled	Thor process	43-7	6.5	-	17.2
(g)	As (c), ball-milled	Pepsin hydrolysis	44-2‡	6-2‡	6-9‡	*******
(h)	Lining of shells of common lobster*	Pepsin hydrolysis	44-5	6-7	6-5	
		Mean values (a, c-h)	44.3	6-5	6-8	second.
		Data given by Meyer and Wehrli ¹ §	46-8	7.09	6-83	20-47
	Substan	NCE		CALCUL	ATED	
Poly-	N-acetylglucosamine (I) (CaH13O	$(N)_m$	47-3	6-4	6-9	21.2
Polyg	lucosamine (chitosan) (II) (C.H.,	$O_4N)_n$	44-7	6-8	8-7	0
82.5%	$I + 12.5\% II + 5\% H_2O$ (by	wt.)	44-6	6-5	6-8	17-5
Polyg	lucose (C ₆ H ₁₀ O ₈) ₈		44-5	6-25	0.	0

· Ash-free.

† Product azeotropically distilled with benzene.

Corrected for ash contents (7.5% and 6.3% respectively).

§ Ash 0.5%.

rinsed with water, dried in air at 105°c., then allowed to condition in air for 24 hr., and stored in a stoppered bottle until required. The material at this stage was colourless. Some preparations were reduced to a fine powder by grinding for 4 hr. in a rubber-lined ball mill with silica balls, and the powder was then washed with water, ethanol, and ether in succession, and dried at 50°c. for 2 days. Most of a sample prepared in this way was found to pass a 200-mesh sieve. Before use it was conditioned in air at room temperature for 7 days. The ash content, insoluble in dilute mineral acids, gave a positive reaction in the molybdenum blue test for silica, and contained a trace of iron. The iron is probably a natural impurity, but the silica is adventitious. For analysis, the material was dehydrated, by soaking first in ethanol and then in ether, dried at 50°c. in air, and finally dried in vacuo over phosphorus pentoxide at 110°c. for 36 hr. The normal moisture content (calculated by drying to constant weight at 105°c.) is about 7.5-9.0%, varying, of course, with the atmospheric conditions. All data in this paper are corrected for the weight of "anhydrous" chitin.

For enzyme removal of protein the treatment employed by Burgess ¹⁴ for degrading wool with trypsin and pepsin was used. The shells (1 g.), broken to ca. \(\frac{1}{4}\) in., were incubated for periods of up to 8 days at 37°c. with 0.5% pepsin solution (5 c.c.) and added pH 1.4 buffer solution (5 c.c.), then washed well in hot water, treated with cold 5% hydrochloric acid solution, again washed well in water, decolorised by acetone extraction, washed with ether, and dried.

ELEMENTARY ANALYSIS

Microanalyses were made by Dr. Weiler and Dr. Strauss, Oxford, and by Dr. A. C. Syme, of this College. The acetyl value was determined after oxidation with chromic oxide.

SOLUTES

Purified commercial or laboratory-prepared materials were used, dissolved in distilled water.

ANALYSES OF SOLUTES

The colourless acids were determined volumetrically, and the coloured solutes absorptiometrically on either a Hilger Spekker photoelectric absorptiometer or a Unicam SP 500 photoelectric spectrophotometer; pH values were measured with a glass electrode.

ADSORPTION TEST PROCEDURE

The chitin samples (0·1 g.) were sealed in glass tubes in presence of 20 c.c. of the appropriate solutions, and tumbled at ca. 35 r.p.m. under water in the thermostat; the apparatus has been described elsewhere ¹⁵.

Preliminary rate measurements showed that all acids reached equilibrium with chitin in under 3 hr., but as a precaution the tests were continued for up to about 12 hr. As a combined check for equilibration and absence of decomposition of solute, the coloured solutions were examined spectrophotometrically before and after the adsorption procedure, and also two duplicate tubes containing the weakest solutions and two containing the strongest were included, and one of each was opened and the contents were analysed after about 3 hr. and 6 hr. from the start.

TEST FOR DEACETYLATION BY ACID

A test was made to discover whether deacetylation is liable to occur on treating chitin with mineral acid for prolonged periods. The nitrogen

content of the material (N = 7.1% originally) immersed in aqueous hydrochloric acid at 60° c. for 24 hr. fell to 6.8, 4.9, and 3.7% (C 45.5, 45.6, and 41.0%; H 6.6, 6.6, and 6.3%) when the final pH values were 2.5 and 2.0 or the initial concentration 2N. respectively. It thus appears that in hot solutions with pH values < 2.5 the acetamido group is removed, ammonia and acetic acid are lost, and some oxidation occurs. In the normal preparation of the starting material, however, the acid treatment is carried out cold.

TEST FOR DIFFERENCES IN ADSORPTIVE PROPERTIES BETWEEN GROUND AND UNGROUND CHITIN

Two samples of chitin, both prepared by Thor's method, one being merely broken into 1 in. flakes, and the other finely ground in an agate mortar for 6 hr., showed a considerable difference in equilibrium adsorptive properties (Fig. 1), for whereas both adsorbed hydrochloric acid to the same extent, their adsorption of sulphuric acid was different. On powdered chitin, the two acids had the same adsorption, but on the flakes sulphuric acid was adsorbed much less than hydrochloric acid. These differences appeared to be quite real and reproducible. They can be attributed to physical disturbances in the crystalline structure of chitin caused by the heat or mechanical action of grinding, which appears to reduce the crystallinity of the substrate.

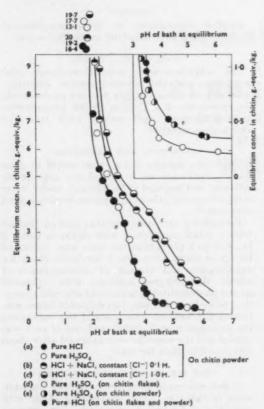


Fig. 1- Adsorption of Mineral Acids by Chitin at 50°c.

All the remaining adsorption tests in the present work were made on flaked chitin.

Molecular dimensions were measured by use of Catalin (Stuart-type) models.

Discussion

CONSTITUTION OF CHITIN

The constitution of chitin has been a subject of frequent investigation over a very long period, though the suggestion made by Ledderhose 16 as long ago as the 1870s, that it is a compound of aminoglucose and acetic acid, is substantially that now accepted; as mentioned above, it is now considered to be poly-N-acetylglucosamine. Meyer and Wehrli's X-ray analysis 7 helped to establish its structure, which has since been studied in greater detail by Darmon and Rudall 10, using X-ray and polarised infrared spectra. From time to time, however, doubts have been expressed about the correctness of the stated constitution; e.g. in 1909 Irvine 17, following polarimetric measurement of acid hydrolysates, suggested that one in four of the glucosamine units is unacetylated, and Morgulis 18 found that 12.5% of the nitrogen was more resistant than the remainder to hydrolysis by sulphuric acid. Darmon and Rudall 10 found evidence of two differently arranged acetamido groups in chitin.

A search of the literature has revealed very few elementary analyses of the substance, and the only complete analysis made in recent years appears to be that of Meyer and Wehrli 7 (see Table II)*. This is a single analysis; the acetyl value corresponds to 96% acetylation, but the C and H values are not in good agreement with this.

Darmon and Rudall ¹⁰ say of chitin that the main test of purity is the nitrogen content, which should be 6·89%, but that this value is seldom attained; material prepared by them from insect cuticles had nitrogen contents of about 6·4%, but that from lobster tendon, they reported, approached nearer to the theoretical value. The present results (discussed below) suggest, however, that the nitrogen content alone may not be an infallible guide to homogeneity.

In view of the need to correlate the results of adsorption tests with the constitution of the substrate, and the slight uncertainty of this constitution, a series of elementary analyses on material prepared by the various procedures described in the Experimental Section above has been carried out, and the details are set out in Table II. It will be seen that the analytical figures for the various preparations do not correspond with that for poly-N-acetylglucosamine. The most reasonable assumption to make to account for this is that a small proportion of the amino groups are unacetylated †. This is not inconsistent with the infrared data, because an inspection of Darmon and Rudall's infrared spectra of chitin and its deacetylation products shows

[•] Since this paper was submitted, Foster and Hackman ¹⁹ have described the separation of a chitin preparation from edible crab cuticle by means of ethylenediaminetetra-acetic acid. The product contained protein (probably < 5%). The elementary analysis (corrected for ash) was C 43-5, H 6-6, N 7-6%.</p>

[†] It was found impracticable to make an analysis for free amino groups on the products used here.

a slight indication of the presence of deacetylated product in chitin. Yet, as will readily be seen by an inspection of the data given, no mixture of the two materials which would be present on this assumption, i.e. the acetamido and the amino derivatives, can give the analysis found, nor could any mixture containing material in which the acetamido group is entirely absent (i.e. polyglucose). The analytical data can, however, be accounted for if some firmly bound water is also present. By testing all possible variations of the resulting three-component mixture, one composition can be found to give a theoretical analysis identical, within experimental error, with the present data for carbon, hydrogen, nitrogen, and the acetyl group. This mixture (Table II) is (in parts by weight)—

Poly-N-acetylglucosamine 82·5 Polyglucosamine 12·5 Water 5·0

The high proportion of water, not removable by normal heating processes, can be justified as follows. Chitin is known to be highly crystalline. Wool, which has low crystallinity (about 10-15% inaccessible to deuterium oxide 20), retains about 0.5-1.0% of bound water after drying, so that a bound water content for chitin of 5% might correspond with a crystallinity of, say, 80%, a figure which is not inconsistent with some of the adsorption data to be described below. In air-dry chitin there is also, of course, a proportion of labile adsorbed water which varies with the state of the atmosphere; this is normally 7.5-9.0% by weight. In all the adsorption data given below correction has been made for this moisture, but not for the postulated bound water.

The free amino groups might be present in the original material or they might be formed by deacetylation during the separation processes, i.e. in either the alkali or the acid treatment. It seems unlikely, however, that they can be formed in these treatments, because (a) the pepsin-treated sample, which has not received alkaline treatment, has not a significantly different analysis from the others, and (b) the effect of acid is rather to reduce the nitrogen content (see Experimental Section above), which means that it hydrolyses off the whole acetamido group and does not leave free amino groups. It must therefore be supposed, in the absence of evidence to the contrary, that the chitin in its normal state has about one glucosamine residue for every seven N-acetylglucosamine groups.

ADSORPTION OF INORGANIC ACIDS

Titration curves of finely ground chitin were determined with hydrochloric acid in presence and absence of sodium or potassium chloride of constant molarity, and with sulphuric acid (Fig. 1).

The constitution assumed for the substrate corresponds with a maximum acid-binding capacity of about 4.8 g.-equiv./kg., i.e. with 4.18 and 0.62 equiv. being taken up by the acetamido and the free amino groups respectively. The titration curve does in fact appear to be reaching a maximum between 4 and 5 equiv./kg. at about

pH 2, but at lower pH values a sudden further rise in adsorption occurs, and proceeds without apparent limit. The acid titration curve for nylons1 shows a similar steep rise, without maximum, at low pH values, but the highest value obtained in that curve is well below that required to combine with all the amide groups. In the present case, of course, once the acetamido and amino groups are saturated, no other acid-combining groups are present, unless the ether groups form oxonium salts. Even if this were so, the total quantity of acid adsorbed could not be accounted for. The results are reproducible, and there appears to be little chance of error in the simple titration method used for analysis. The substrate becomes gelatinous in the strongly acid solutions, but the acid adsorption cannot be accounted for by occlusion of the external liquid by the gel, because occlusion would not affect the titration values. It must be supposed that at very low pH values the acids are taken up in some unidentified form, perhaps by hydrogen bonding of unionised acid molecules at the hydroxy groups or by physical adsorption of some form of micelles of associated unionised molecules.

The reaction of mineral acid with the acetamido groups in chitin may be compared with its reaction with wool; thus the following equations represent combination of an acid HX with (i) wool and (ii) chitin—

$$Wl \mathring{N}H_3 \} \widetilde{O}OC \cdot Wl \xrightarrow{H^+} Wl \mathring{N}H_3 + HOOC \cdot Wl$$

$$\xrightarrow{X^-} Wl \mathring{N}H_3 \} X^- + HOOC \cdot Wl \qquad (i)$$

$$\text{Ch-NH-CO-CH}_{9} \xrightarrow{\text{H}^{+}} \text{Ch-NH}_{9}\text{-CO-CH}_{9}$$

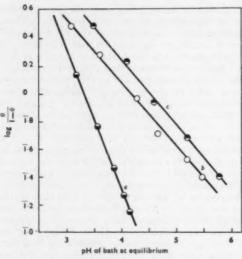
$$\xrightarrow{\text{X}^{-}} \text{Ch-NH}_{9}\text{-CO-CH}_{9}\text{X}^{-} \qquad \text{(ii)}$$

(WI and Ch = residues of wool and chitin molecular chains).

Until dissociated by the entering hydrogen ions, nearly all the basic amino and the acidic groups in wool are combined as salt links, and thus probably do not act as separate ionic entities. When the fibre is saturated with acid no salt links are present, and the only ionic centres are the charged amino groups. Thus in wool and in chitin, after adsorption of the hydrogen ion, the anion is adsorbed at a surface whose only effective ionic sites are singly charged cations. It may therefore be assumed that comparison of the two systems by similar quantitative means is justified.

The quantitative treatment of acid adsorption by wool is well known 21-25: thus in the Gilbert-Rideal hypothesis it is assumed that adsorbed anions are located at independent specific sites, but in the hypothesis which treats the system as a Donnan membrane the adsorbed anions are not assumed to be attached to specific sites. Each hypothesis satisfies some, but not all, of the experimental facts. In the Gilbert-Rideal hypothesis, the activity of the hydrogen ions in the fibre is assumed to be $\theta/(1-\theta)$, where θ = the fraction of the available sites occupied; the affinity of hydrochloric acid is thus given by the expression—

$$-\frac{1}{2}(\Delta\mu_{\rm H}^{\circ} + \Delta\mu_{\rm Cl}^{\circ}) = 2.3 \ RT \log \left[\theta/(1-\theta)\right] + pH + pCl$$
 (iii)



(a) Acid alone

(b) Constant chloride concn. (0-1 M.)

(c) Constant chloride concn. (I-0 M.)

Fig. 2— Hydrochloric Acid Adsorption Data (specific site interaction hypothesis)

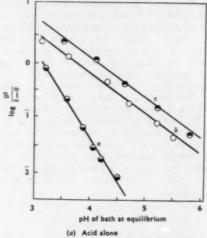
from which it follows that there should be a linear relationship between log $[\theta/(1-\theta)]$ and pH, as indeed there is for wool. Fig. 2 shows that there is a linear relationship for chitin also, with slopes of -1.0 (acid alone), -0.46 (0.1N-NaCl), and -0.48 (N-NaCl), compared with the theoretical values of -1.0, -0.5, and -0.5 respectively required by equation (iii) (cf. the corresponding values ²⁴ for wool: -0.88, -0.50, -0.50). The affinity values for hydrochloric acid calculated from (iii) for wool, however, are virtually constant at all the acid or chloride concentrations, whereas here they show a downward trend with increase in either acid or chloride concentration*.

The differences between the two substrates may be a result of differences in the accessibilities of sites to the anions. In wool the proportion of the total sites available for reaction is determined solely by their degree of ionisation, which in turn is a function of the pH of the solution. Chitin is apparently much more crystalline than wool, so that the accessibility of its acetamido (and amino) soups may well be restricted by the mechanical difficulty of penetration of the structure, and at any given pH large anions, which would be expected to have higher affinity than the chloride ion, are adsorbed much less than the latter, because they cannot reach as many sites. It is therefore possible that the chloride anion also has no! access to as high a proportion of the theoretically possible sites as has the hydrogen ion, and thus with added chloride the increase in adsorption is less than theoretically predicted.

The expression for the affinity of hydrochloric acid for wool considered as a Donnan membrane

$$-\Delta \mu_{\rm H}^{\circ} = 2.3 \, RT \, (\log \frac{S_{\rm H}}{V} + \log \frac{\theta^2}{1-\theta} + {\rm pH} + {\rm pCl})$$
 (iv)

 This trend is much greater than can apparently be accounted for by neglect of activity coefficients ^{cf. 24}.



(b) Constant chloride concn. (0-1 M.)

(c) Constant chloride concn. (1-0 m.)

Fig. 3— Hydrochloric Acid Adsorption Data for Chitin (Donnan membrane adsorption hypothesis)

where $S_{\rm H}$ is the saturation adsorption value, and V is the volume of internal solution in the substrate. Plots of $\log \left[\theta^2/(1-\theta) \right]$ against pH (Fig. 3) are linear, as required by this equation, but the slopes for adsorption from solutions containing acid alone, and those with constant chloride ion concentration (0·1N. and 1·0N.), are respectively $-1\cdot6$ and $-0\cdot72$, compared with the theoretical values of $-2\cdot0$ and $-1\cdot0$ (cf. the corresponding value of $-1\cdot4$ for acid alone on wool ²⁴). The affinity values for chitin cannot be calculated from equation (iv) in absence of a value for V.

As already stated, chitin is probably highly crystalline. On general considerations the actual state of the ions in any substrate may be considered to lie between the two suggested extremes of complete fixation at cationic sites or completely unlocalised solvation, so that the more highly crystalline the substrate the more restricted should be the mobility of the anions in the internal aqueous phase, and the nearer should the conditions approach to those assumed in the localised-site theory.

ADSORPTION OF ORGANIC ACIDS

Adsorption tests were made with a variety of organic acids, titration curves (Fig. 4) being plotted showing the amount adsorbed from solutions over a range of equilibrium pH values, obtained by variation of the concentration of the acid. Table III gives some comparative data showing the variation in amounts of several organic and two inorganic acids adsorbed at two equilibrium pH values, and some of the data are plotted in Fig. 5 in comparison with corresponding data for wool ²⁶.

It is clear that there is a fundamental difference between the data for the two substrates. For wool the general tendency appears to be for adsorption to rise with the molecular size of the acid. For chitin the situation is rather more complex, and two opposing factors seem to be operating: broadly,

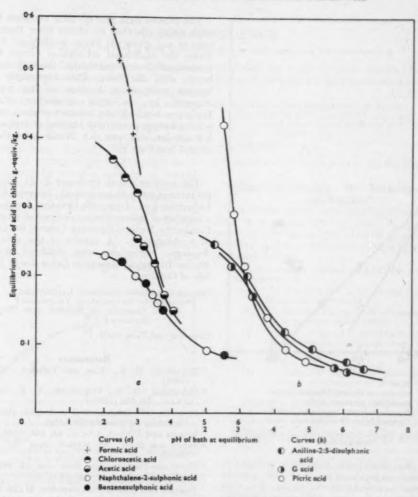


Fig. 4- Adsorption Isotherms for Organic Acids on Chitin

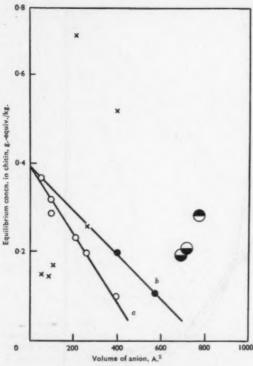
TABLE III

Advertion of Acids by Unground Chitin and by Woo

Adsorption of Acids	by Unground Chiti	n and by t	Vool	
Acid	Approx. Vol. of Anion*	Amount adsorbed (m-moles/kg.)		
	(A. ³)	Chitin (50°c.)	3·0 Wool (0°c.)†	pH 4-5 Chitin (50°c.)
Hydrochlorie	30	3870	170	450
Formie	55	385	140	-
Acetic	90	255	160	
Chloroacetic	120	320	165	-
Sulphurie	110	3870	0110	150
Picrie	210	235	660	85
Benzenesulphonic	260	200	265	100
Aniline-2:5-disulphonic	390	100		55
Naphthalene-2-sulphonic	400	195	500	-
2-Naphthol-6:8-disulphonic (G acid)	570	105	parent.	50

[·] Volume of enclosing rectangular box.

[†] Approximate values, by interpolation from data of Steinhardt et al. 38.



× Wool

- (a) O Aliphatic and benzene compounds (chitin)
- (b) Naphthalene compounds (chitin)
 - Phenylazonaphthalene compounds (chitin)
 - Naphthylazonaphthalene compound (chitin)

Fig. 5— Relation between Volume of Anion and Adsorption on Chitin and Wool

it appears that adsorption rises with increase in length of the conjugate chain, but falls linearly with increase in volume of the anion. The first effect is well known to occur with anionic dyes on cellulose, and it seems reasonable to attribute it to the same cause in cellulose and in chitin, viz. the increase in physical or dispersive force between the π -electron system of the anion and the planar contour of the water-surrounded molecular chain of these substrates*.

The fall in adsorption with volume of the anion must be a purely mechanical effect arising from the highly crystalline structure of chitin. Fig. 5 shows that the maximum adsorption of a small organic anion at pH 3 is about 400 m-equiv./kg., which is about 10% of the amount of hydrochloric acid taken up at the same pH value (see Fig. 1). The facts are consistent with the existence in wet chitin of pores in a range of different sizes; the largest pores can (at pH 3) accommodate any anions with volumes up to about 500 a.3. When the attraction of the anion for chitin is increased by lengthening its conjugate chain, it can apparently force its way into other parts of the chitin structure, and thus in effect create larger pores.

• It will be observed from Fig. 5 that the benzenoid anions lie on the same line as the aliphatic ones, so that the non-polar attraction of a single aromatic nucleus for the polyglucosidic chains in water must be very low. This is consistent with the non-adsorption of benzenoid solutes (e.g. benzenesulphonic acid, phenol) byteclulose from water. The present data give no clear evidence of weak acids being adsorbed by chitin more than strong ones at any given pH value, as they are by wool, where the additional adsorption occurs through undissociated acid molecules forming hydrogen bonds with the fibre. This apparently cannot happen with chitin because of the restriction imposed by its rigid crystalline structure. Hydrogen-bond affinity becomes evident only with solutes having powerfully binding groups and very low ion-ion and van der Waals attraction, e.g. phenol (see Part V²⁷).

The authors thank Professor P. D. Ritchie for his interest and encouragement; Imperial Chemical Industries Ltd., Dyestuffs Division, for the gift of a number of intermediates; and one of them (M.L.) thanks the Medical Research Council for the grant of a scholarship. A supply of the carapace of Nephrops norvegicus was obtained from the Marine Biological Research Laboratory, Millport,

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(Received 14th June 1957)

Isle of Cumbrae.

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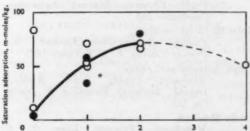
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CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

The Mechanism of the Adsorption of Disperse Dyes by Cellulose Acetate

Daruwalla and Limaye ¹ have reported saturation adsorption data for various anthraquinone and azo disperse dyes on cellulose acetate, and conclude that, because in selected cases there appeared to be no correlation between saturation adsorption and hydrogen-bond capacity of the dye, the hypothesis of a hydrogen-bond adsorption mechanism, proposed by various authors, is invalid. These data, however, give much more interesting results than the authors themselves claim; e.g. Fig. 1 shows that there is in fact a relation between the number of hydrogen atoms in the dye available for bonding with the fibre and the saturation adsorption. This appears to be a good confirmation that adsorption is due mainly to Dye–OH . . . Fibre and Dye–NH . . . Fibre hydrogen bonds.



No. of H stoms in dye molecule available for bonding with cellulose acetate

- Axo dyes
- Anthraquinone dyes (Celliton Blue 3G omitted because of uncertainty of constitution)

Fig. 2— Relation between Number of -NH... or -OH... Intermolecular Bonds formed by Disperse Dyes and their Saturation Adsorption on Cellulose Acetate

* It is assumed that the two hydroxy groups in the bis- β -hydroxy-ethylaniline dye are chelated, leaving one free hydrogen atom.

A very similar relation to that shown in Fig. 1 is obtained by plotting water solubility instead of saturation adsorption. Saturation adsorption rises roughly proportionately with solubility in water. High adsorption in the fibre is therefore not due to any tendency for the dye to escape from the water phase. This is consistent with "solid solution", i.e. adsorption in regions inaccessible to water.

There are some exceptions to the relation. An examination of these exceptions, and of data

published by other authors, shows that several factors determine the maximum adsorption.

C. H. Gilles

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14th July 1958

¹ Daruwalla and Limaye, J.S.D.C., 73, 464 (June 1958).

"Fibre Microscopy—Its Technique and Application"

The encyclopaedic aims attributed to the above book in a recent review make it necessary to point out that the ground which the author set out to cover is clearly defined in the preface. Here it is stated that—

- . . . The strictly limited aims of the work can be summarized as:
 - To describe the most efficient use of the compound microscope.
 - (2) To describe the methods of preparing fibres for microscopic examination.
 - (3) To give, in broad eutline, an account of the more advanced techniques of modern microscopy and their application to the study of fibres.
 - (4) To indicate, inter alia, examples of the technical applications of fibre microscopy in industrial practice.

Extensive references to the literature have been given so that where desired the reader can pursue his studies in greater detail. It is realized that in certain fields, fibre research has made further progress while this book was being written. The references and bibliographies, however, indicate the main schools of research and the journals in which their results are published. It should be relatively easy, therefore, to make contact with the front line of advance.

... Beginners are advised to read Chapters 1, 2, 3 and 6 and then turn to Part II. After this study of the compound microscope and its use in fibre examination attention can be turned to the more advanced techniques described in Chapters 4 and 5, and 7 to 10.

The price of the book (50s.), which the review considers to be excessive, is closely related to present-day costs of blockmaking. To have added to the existing 210 figures, as is suggested in the review, would have increased the cost of production still further.

J. L. STOVES

THE LABORATORIES 61 GRANGE ROAD LONDON S.E.1

24th June 1958

1 J.S.D.C., 74, 489 (June 1958).

Notes

H.R.H. The Duke of Edinburgh, K.G., K.T. Patron of 1959 International Congress

The Society of Dyers and Colourists, as the host member society, is greatly honoured to announce that His Royal Highness The Duke of Edinburgh has consented to be Patron of the Congress of the International Federation of Associations of Textile Chemists and Colourists, to be held in London during 16-18th September 1959.

Meetings of Council and Committees August

Council-No meeting

Publications-19th

Colour Index Editorial Panel-8th and 20th Washing Fastness Subcommittee-15th.

Deaths

We regret to report the loss by death of Mr. J. E. Eastwood, Mr. George D. Halley, Mr. Ho Iu Tim, and Mr. E. M. Walker.

Election of Fellows and Associates

At the meeting of Council held on 3rd September 1958 the following elections were made-

Fellows

José Sánchez Cegarra

Sabadell, near Barcelona, Spain; Director and Professor, Escuela Técnica Superior de Ingenieros Industriales, Sección Textil, Tarrasa, consultant textile engineer.

John Greenwood

Manchester; Dyehouse Manager, R. P. Robert Edward Todd Lawson & Sons Ltd.

Stanley Shaw

Pontypool, Monmouthshire; Senior Technical Representative, British Spinners Ltd.

Ernest Yeadon

Westerly, Rhode Island, U.S.A.; Technical Director, Bradford Dyeing Association Inc.

Associates

BRANCH 1-TINCTORIAL TECHNOLOGY AS APPLIED TO TEXTILES

Kenneth Crossland

Works Yorkshire; Assistant Batley, Chemist, J. T. & J. Taylor Ltd.

John Dickson

Heanor, Derbyshire; Manager of Finishing Department, I. & R. Morley Ltd.

Hugh Malcolm Duff

Dungannon, County Tyrone, Northern Ireland; Assistant Colourist, Stevenson & Son Ltd.

Peter Ginns

Coventry, Warwickshire; Textile Chemist, Dyehouse Laboratory, Courtailds Ltd.

John Keith Hanson

Bradford, Yorkshire; Research Laboratory Assistant, The Bradford Dyers' Association Ltd.

Neil Shackleton Hemingway

Norden, Rochdale, Lancashire; Chemist, Rainshore Bleaching & Dyeing Co. Ltd.

Ronnie Lawson

Bradford, Yorkshire; Technical Dyer, The Bradford Dyers' Association Ltd.

Keith David McKelvie

Bradford, Yorkshire; Apprentice Dyer, The Bradford Dyers' Association Ltd. (Airedale Dyeing Co. Ltd., Keighley)

Ildo Emil Pensa

Flixecourt (Somme), France; Technical Management, Saint Frères S.A.

Arnold Samuels

Droylsden, near Manchester; Assistant Textile Chemist, Research Laboratory, Courtaulds Ltd.

John Mirkland Scadlock

Paisley, Renfrewshire, Scotland; Wet Process Trainee, J. & P. Coats Ltd.

Hugh John Stewart Shane

Banbridge, County Down, Northern Ireland; Ballievey Bleaching & Dyeing Co. Ltd.

Leslie Shedlock

Manchester; Foreman Dyer, T. E. Marchington & Co. Ltd.

John Todd

Manchester; Chief Chemist, Mark Fletcher & Sons Ltd.

Bradford, Yorkshire; Dyer, The Bradford Dyers' Association Ltd. (Hunsworth Dyeing Co. Ltd.)

Light Fastness Subcommittee

Unfortunately the name of F. W. LINDLEY, Ph.D., was omitted from the list of members published in the July 1958 issue of the Journal (74, 512).

The Perkin Centenary Trust Awards for 1958-1959

The Perkin Centenary Fellowship has been awarded to Mr. Dennis Balmforth, B.Sc., A.R.I.C., of Coventry, and will be tenable from 1st October 1958 in the Department of Colour Chemistry at the University of Leeds, where Mr. Balmforth intends to carry out research on a problem related to the application of dyes.

Perkin Centenary Scholarships have been awarded to Mr. Brian Higginbottom of Bolton, tenable at the College of Technology, Loughborough, and to Mr. W. K. McCourt of Belfast, tenable at Queen's University, Belfast.

Arrangements have also been made for a Perkin Exchange Lectureship whereby short visits will be exchanged between Professor A. J. Birch, F.R.S., of the University of Manchester, and Professor V. Prelog, of the Federal Polytechnic in Zurich. Professor Birch is visiting Zurich in September 1958, and it is hoped that Professor Prelog will return the visit in 1959. Each will deliver one or more lectures during his visit, which will last about two or three weeks.

The Perkin Centenary Trust Awards for 1959-1960

The Trustees invite applications for the following awards for the academic year 1959-1960—

THE PERKIN CENTENARY FELLOWSHIP

This is offered for one or two years to a graduate for the purpose of higher study of any subject related to the manufacture or the application of colouring matters. It has a value of not less than £600 per annum, and is tenable, from October 1959, at any university, technical college, or other institution, approved by the Trustees. Applications, on forms available from the Secretary, must be received not later than 1st May 1959.

THE PERKIN CENTENARY SCHOLARSHIP

Two such awards are offered, each for two years starting in October 1959 and renewable at the discretion of the Trustees for one further year, to enable candidates employed in an industrial firm or other institution concerned with the manufacture or the application of colouring matters to receive

an education at a university or a technical college. Each award will have a value of £300 per annum. There is no means test for the award, and a successful candidate is not debarred from receiving the whole ora part of his normal salary from his employers during his tenure of the Scholarship. Applications, on forms available from the Secretary, must be received not later than 1st May 1959.

PERKIN CENTENARY TRUST TRAVEL GRANTS

The Trustees will consider awarding grants to assist teachers concerned with the study of any aspect of the manufacture or the application of colouring matters at any appropriate university, technical college, or other institution, to make short visits to comparable institutions overseas in order to increase their knowledge of the subject. Applications must be received by the Secretary not later than 1st May 1959. These should contain as much detail as possible about the objects and the duration of the intended visit and the amount of grant required. They should also be accompanied by the recommendation of the professor or head of the department in which the applicant is working. These grants will, for the year 1959-1960, take the place of the Perkin Exchange Lectureships advertised in the two previous years.

Enquiries relating to the above awards should be addressed to The Secretary, The Perkin Centenary Trust, c/o The Chemical Society, Burlington House, London W.1.

New Books and Publications

The Manufacture of Lakes and Precipitated Pigments

By A. W. C. Harrison. Revised by J. Stewart Remington and Wilfred Francis. 2nd edition 1957. Pp. xx + 284. London: Leonard Hill (Books) Ltd. Price, 30s. 0d.

The dye industry, like many other specialised branches of chemical technology, has been badly served with practical textbooks. After the last war, however, an immense amount of accurate up-to-date information was published in the B.I.O.S. and F.I.A.T. reports, and could have been utilised by any post-war author. It is therefore very disappointing to find that in Remington and Francis's revision of Harrison's book no use whatever has been made of what they themselves call "One considerable source of knowledge on the subject of pigments", apart from inclusion of references, in rather haphazard manner, in bibliographies at the ends of chapters and in an appendix.

In the preface to the second edition the authors say: "... we have left untouched as much of the original as possible, altering only those parts which are badly out of date...". The truth of the first part of this statement is only too obvious on comparing the second with the first edition, but one looks in vain for any real modernisation of the

text. Perhaps the insertion in a section on filter presses (p. 48) of the solitary new phrase "though stainless steel presses are now available", and the substitution on p. 50 of "nylon" for "cloth" in a section on screw presses, which should have been deleted anyway, are to be taken as revision.

When published in 1930 Harrison's book was hardly up to date and it contained many inaccuracies. The second edition in 1957 is in many respects 27 more years out of date, the errors have been retained, and a number of misprints have been added for good measure.

Of the new matter added by the present authors the sections on light and colour in relation to pigments and on the use of B.S. 1006 are useful additions. That on water softening, whilst not irrelevant, is not of prime importance, and that on fuel economy has been better dealt with in Ministry of Fuel and Power publications.

If a thoroughgoing revision had been carried out, correcting errors, replacing Harrison's making formulae by examples from German practice, and bringing the section on plant and equipment up to date, then a book of some use to students and entrants to the pigment industries would have resulted. In its present form it cannot be recommended. For those seeking information on

pigments, reference can be made to two of the sources quoted by Remington and Francis, viz. B.I.O.S. 1661 (German Organic Pigments and Lake Dyestuffs) and B.I.O.S. 1402 (The German Manufacture of Certain Inorganic Pigments). A digest of some of the data in these two reports alone would have been of greater value than the present publication. D. M. STEAD

Guida dei Principali Prodotti Chimice Volume Secondo

By Cesari Ferri. Pp. xv + 650. Bologna: Nicola Zanichelli Editore. 1958. Price, 7,000 lire.

Whereas the first volume dealt with the main groups of industrial chemical products (cf. J.S.D.C., 72, 173 (1956)), this second volume lists individual products, giving under each—commercial names, chemical and physical data for the pure material (molecular weight, boiling point, solubility, etc.), data relating to the industrial product (quality, impurities, method of packaging, etc.), method of manufacture, reactions of industrial interest, and uses. Inorganic products (272 pp.) are arranged alphabetically-from acqua to zirconio oseidomainly under their principal elements; organic products (376 pp.) are listed in straightforward alphabetical order—from abasina to zucchero.

Since colorants are specifically excluded from the organic section, this volume will be of limited interest to readers of this Journal. However, important mordants and intermediates are included, and the book will serve as a primer of chemical Italian to those e.g. who are interested in puzzling out the relevance of chetoesametilen-

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., 68, 23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index

BP 796,423

I-PLANT; MACHINERY; BUILDINGS

PATENTS

Alloys resistant to Chlorite Bleaching Liquors

Deutsche Gold- und Silber-Scheideanstalt An alloy of 10–25% Cr, 0–10% W, 0–2% Mn, 0–1% C, 0–1% Si, \ll 40% Ni and \ll 4-5% each of Fe and Mo, the Fe and Mo content being less than that of the Ni, is almost completely resistant to acid chlorite solutions even of strengths greater than those usual for bleaching.

Continuous Liquid Treatment of Artificial Threads BP 796,367

Washing Raw Wool and Similar Fibrous Materials BP 796,021 Aktiebolaget Separator

The wool together with the detergent liquor is fed through a passage between two opposite surfaces. One of the surfaces is movable relative to the other and forms part of an endless path through which the liquor flows, the wool being fed through the slot by pins or elastic wires on the movable surface. The detergent liquor and the wool are brought to pass together through the slot under such friction conditions that the wool is distributed in the liquor as a suspension. The wool passes through the machine in < 1 min., preferably < 1 sec. Thus the moving surface may be a cylinder studded with pins which revolves within a casing which forms the opposite and stationary surface.

Continuous Bleaching and Dyeing Machines

E. M. J. Larcher The material passes through a U-shaped tank in which liquor is circulated from the top of the inlet arm to the bottom of the tank. The outlet arm is narrower than the inlet arm. The loop of the U joining the two arms consists of two parts, one of which decreases in width from that of the inlet to that of the outlet arm. A pump in this part circulates the liquor to the top of the inlet arm at such a rate that the liquor in the lower part of the tank settles at a level the vertical distance of which from the level of the liquor in the upper part of the tank provides a head equal to the pressure drop of the liquor incurred during its passage through the goods.

C.O.C.

Dyeing Machine W. Watkins

Dye liquor is confined in the bight between two padding rollers. Fabric passes through this liquor and through the nip into a heating chamber. This enables the use of very short liquors with frequent changes in either the colour or the class of dye to be used.

Dyeing or other Wet Processing Plant

Freeman, Taylor Machines BP 796,328

A dyeing or other wet processing plant comprises at least two machines each having in its liquor tank a propeller for circulating the liquor. The machines are directly flowconnected by conduits so that some of the liquor impelled by the propeller of each machine is branched from the compression side of the propeller in the one machine to the suction side of the propeller in the other machine and vice versa. By incorporating in the plant machines of different sizes it is possible to cope with and treat simultaneously under identical conditions separate lots of goods divided out from a larger quantity.

Pressure Rollers

British Cotton Industry Research Assocn. A pressure roller consists of a non-resilient core having a resilient cover whose thickness and/or elastic modulus vary in a pre-determined manner along the length of the roller. variation is so chosen that the resulting variation in the hardness of the cover is such that bowing of the collar or of its complementary roller under pressure which would disturb the uniformity of pressure distribution, is at least in part compensated for. This reduces the variation of pressure distribution along the roller.

Roller Felting Machines William Bywater There are two sets of rollers, in which each roller of at least one of the sets moves reciprocally parallel to its axis and/or oscillates about its axis in addition to rotating in the normal manner, mounted in two rows one above another. The rollers in one row are staggered relatively to the rollers in the other row. The rollers of one set, usually the lower set, are adjustable vertically so that pressure may be applied to cloth or felt passing between the two rows of rollers. The vertically adjustable rollers are mounted in groups on side frame members supported by platforms which are raised and lowered by suitable

Drying Warps, Fabrics, etc. Comptoir de l'Industrie Cotonnière, Etablissements Boussac

The warp or fabric passes through a preheating chamber and then over a drying cylinder. The air in the chamber containing the drying cylinder passes into the preheating chamber, some of it passing to the outer atmosphere through an exhaust placed between the drum and the proheating chamber. The air in the preheating chamber is sent back over heating elements to the cylinder chamber, fresh air to compreheate for that best through the order. fresh air to compensate for that lost through the exhaust being admitted into the cylinder chamber.

Sept. 1968 Cloth Drying

BP 795,314 The cloth is carried on one or more horizontal endless

band conveyors between upper and lower air ducts or casings each having an opening from which warm air is directed onto the cloth.

Continuously Heating Fabric, Paper, Films, Foils, etc. USP 2,800,725 A machine in which the fabric or the like passes over

heating surfaces which are automatically lowered if passage of the fabric stops. C.O.C.

Controlling the Tension of Fabrics in Raising Machines A. Monforts BP 796,729

Nylon Hose Preboarding L. Girling and M. J. Andrew

BP 794,718 Groups of hosiery forms vertically mounted on form carriages are introduced into a vertical pressure chamber carriages are internally slidable doors. The chamber accommodates two carriages introduced from opposite sides enabling two operators to work simultaneously the four carriages provided. G.E.K.

Hosiery Inspection Form

Textile Machine Works An expanding form from which the hose are stripped by drawing the toe down a central tube aided by air currents. The hose are turned during the operation and emerge from the base of the form for collection.

Garment and Hosiery Pressing Machines BP 796,228 Isaac Braithwaite & Son Engineers

Pelleting Finely-divided Solids such as Carbon Black with Liquids (IV p. 667) Plural Colour Doctor Knife for Use with Textile Printing Machines (IX p. 671)

Bisulphite as an Agent for Binding Free Formaldehyde in Crease-resist Finishing Baths (X p. 671)

II— WATER AND EFFLUENTS

Reducing Textile Waste Problems by Using Starch Substitutes

J. A. McCarthy

Sewage & Ind. Wastes, 28, 334-336 (1956): Chem. Abs., 52, 7710 (10 May 1958)

Use of carboxymethylcellulose, carboxymethylated starch, hydroxymethylcellulose, and various acrylic, vinyl or styrene polymers instead of starch for sizing is sufficiently justified by the reduction in the B.O.D. of the wastes from desizing. C.O.C.

III- CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Textile Auxiliaries

S. Jost

Textil-Rund., 13, 292-304 (May 1958)

Fluorescent brightening, sequestering, delustring, softening, anticrease, flameproofing, antistatic, water-proofing, and various proprietary compounds are tabulated.

Non-ionic Surface-active Agents. II—Synthesis of some Polyoxyethylene Glycol Monohexyl Ethers B. A. Mulley

J.C.S., 2065-2066 (May 1958) The preparation of the title substances with 2-6 ethylene oxide units per mol. is described by the Williamson ether synthesis via the route-

CaHasBr+Na·O·[CHa·CHa·O]a·H $\rightarrow C_6H_{13} \cdot O \cdot [CH_2 \cdot CH_2 \cdot O]_n$

 $\rightleftharpoons \mathrm{C_0H_{13}\text{-}O\text{-}[\mathrm{CH_3}\text{-}\mathrm{CH_2}\text{-}\mathrm{O}]_{\mathrm{H^{-1}}\text{-}\mathrm{CH_2}\text{-}\mathrm{CH_2}\mathrm{C}\mathrm{I}}}$

to minimise the formation of dialkyl ethers, approx. 8 equiv. of glycol were used to one of sodium, so that mainly monosodium deriv, were obtained. These epd. have lipophilic and hydrophilic properties and are useful for the study of non-ionic surface-active agents. The cpd. with 2 and 3 ethylene oxide units severally are almost insol. in water at 20°c., but higher members are sol. and possess a hydrophilic nature which increases with the number of oxide units per mol. The monoethers with n=2 and 3 were obtained from di- and tri-ethylene glycol, and gave the derived chloro epd. with thionyl chloride in presence of pyridine; these condensed readily with the appropriate monosodium deriv. of a glycol to give monoethers with n = 4, 5 and 6.H.H.H.

Benzyl Ethers of o- and p-Phenylphenols

Przemysł Chemiczny, (7), 394-395 (1957):
Polish Tech. Abs., 29, (1), 99 (1958)
By steam distillation of solutions of sodium o- and pphenylphenoxides and benzyldimethylphenylammonium chloride (Leukotrope O), used in molar ratios, benzyl esters of o- and p-phenylphenol, not hitherto described in the literature, were obtained in 90-95% yield. Intermediate products—undissociated salts—are obtained.

Chemical Compounds as Fungicides. I—Certain S-Alkyl, Alkoxyalkyl, and Arylmercury Derivatives of 2-Mercaptobenzothiazole

B. Hetnarski, Z. Eckstein, and T. Urbański

Przemyst Chemiczny, (5), 291-293 (1957): Polish Tech. Abs., 29, (1), 97 (1958)

Several such derivatives were prepared by the reaction of the sodium salt of 2-mercaptobenzothiazole with the corresponding halogen derivatives. Compounds of this showed a strong fungicidal activity toward the strains of microorganisms investigated. The activity of these compounds depended to a high degree on the hydrocarbon group bound to the mercury atom.

C.J.W.H.

PATENTS

Detergents

Eastman Kodak Co. USP 2,798,047

Addition to an anionic or non-ionic detergent of a copolymer of lower N-alkyl-acrylamides and vinyl alcohol prevents redeposition of soil on fibres when they are washed with these detergents.

Stable, Anhydrous NN-Alkylene Urea Compounds

A pure anhydrous alkylene imine, e.g. ethyleneimine, is gradually stirred into an anhydrous aliphatic isocyanate, e.g. octadecyl isocyanate, while cooling externally. The resulting molten material solidifies on cooling to a hard wax-like mass having excellent stability when stored at room temperature. It contains the monomeric alkylene ures even if kept hot for three months. These products can be dissolved in organic solvents or dispersed in water at any desired moment to produce resin finishes for textiles, waterproofing agents for use in dry-cleaning solvents, etc.

Aminoplast Precondensate for Treating Cellulosic Textiles

Courtaulde water-soluble resinous precondensate is A stable, obtained by treating a resin-forming compound containing >1 NH₄ group and in addition the group >N-CX-N (X=0 or >NR; R=H or $>C:N\cdot C$ the other end of which is directly attached to one of the other N atoms) with an epihalogeno hydrin and then with HCHO.

COC Alkoxymethylene Quaternary Ammonium Compositions for improving the Dimensional Stability of Regenerated Cellulose Textiles

Onyx Oil & Chemical Co. USP 2,793,929 An amine is treated with cone. HCl or HBr and the resulting solution evaporated until an anhydrous hydrohalide is obtained. This is then treated with methanol and named is obtained. This is then treated with methanol and paraformaldehyde at pH < 3 at 20–80°c. This yields a composition containing quaternary ammonium halides having a methoxymethyl group as an N-substituent. Fabric containing regenerated cellulose fibres is impregnated with a 5–10% aq. solution of this composition at pH 2–5, dried and baked for 2–10 min. at 145–160°c.

Quaternary Ammonium Derivatives of Unsaturated Esters and Amides and Polymers thereof—Finishing and Auxiliary Agents

Ciba Solutions or aqueous dispersions are obtained by jointly polymerising (1) an ester or amide containing at least one quaternary group bound to the nearest hetero atom by a hydrocarbon bridge of > 1 C with (2) an ethylenically unsaturated compound which if it is aerylonitrile or an homologue of it must not form >50% of the monomers present. The process and products have wide applications; thus the monomers can be polymerised in situ on textiles or other substrate or the polymers can be used for all the usual applications of resins. Use of suitable monomers enables water-repellent finishes for textiles to be obtained, cellulose to be animalised, the wet fastness of direct dyeings to be improved, etc.

C.O.C.

Phosphorus-containing Polyester Resins for making Flame-resistant Paints

Albright & Wilson BP 796,446
Transesterification between a dialkyl phosphite and an aliphatic glycol according to the scheme—

(X = aliphatic hydrocarbon residue; n = whole number) yields products useful as components in making flameresistant coating compositions. Thus a mixture of dimethylphosphite, ethylene glycol and Mg was gently heated in an atmosphere of N. Methyl alcohol began to distil off at 100°c. The mixture was raised over 6 hr. to 200°c. and kept at this temperature until all the volatile material had distilled off first at 15 mm. and finally at 0·1 mm. Hg. The product when cold was a water-white transparent glassy material which would just flow extremely slowly when the container was inverted.

Cold Waving Composition Devon Cold Wave Co.

Devon Cold Wave Co.

A composition for rendering hair more receptive to permanent waving by any of the usual systems comprises a mixture of equal weights a-amino-y-methylmercapto-butyric acid and a-amino-\(\beta\)-hydroxypropionic acid.

Ultraviolet Radiation Absorbing Agents

Compounds of formula—

A·N:N·CXY·H

BP 795,250

(A = benzene having at least one negative substituent; X and Y = same or different, CN, COCH₃, CONH₃ or COO Alk (Alk of < 5 C)) e.g. 2:5-dichlorophenylazo-cyanacetate, when incorporated into synthetic polymers, coating compositions, cosmetics, etc. give good protection against degradation by ultraviolet radiation and have no deletering effects.

Organic Silicate Compounds as Driers for Paints, etc.

Midland Chemical Corpn.

USP 2,798,057
Organic silicate compounds, e.g. ethyl orthosilicate Si(OC₂H₃)₄, when used as driers in coating compositions which are either air-dried or baked prevent the discoloration or yellowing of the paint which occurs when metal driers are used.

C.O.C.

Urethane-Formaldehyde Condensates as Tanning Agents

BASF
The condensates of polyfunctional urethanes, e.g. αδ-butanediurethane, with formaldehyde are tanning agents which can be used in acid or alkaline solutions to yield pale-coloured leather having a very compact, firm handle of adequate softness.

C.O.C.

Pyridine-2:6-dicarboxylic Acids as Sequestering Agents in Photographic Elements or Processing Baths

Gevaert Photo-Producten

Derivatives of pyridine containing a carboxyl group in the 2- and 6-positions of the pyridine ring have very strong sequestering power for Ca and Mg and the chelates formed are exceptionally stable. Their presence during photographic processing causes no disturbing side reactions in the processing baths and has no deleterious effect on the quality of the photographic material or on the appearance of the printed images.

C.O.C.

Printing Pastes, Sizes, Films, etc. (IX p. 671)

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Johann Peter Griess (1829-1888)— Discoverer of Diazo Compounds
W. H. Cliffe

A biographical sketch. In 1858 he discovered that the action of nitrous acid on picramic acid in the cold gave a new type of compound, for which he coined the term "diazo". These reactive diazo compounds could react with other substances to give brilliantly coloured materials, some of which had the valuable property of being able to dye cotton without the use of mordants. Surprisingly, Griess made no financial gains from his work, which laid the foundations of the azo-dye industry.

E.V.T.

Electronic Photoreactions of Organic Dyes

A. N. Terenin Experientia Suppl. 7, 343–355 (1957):

Chem. Abs., 52, 8753 (10 June 1958)
A lecture, 14 references. C.O.C.

Direct Substitution of the Nitro Group in Aromatic Nitro Compounds by Chlorine using Carbon Tetrachloride

A. A. Ponomarenko

Ukrainian Chem. J., 24, 68-72 (Feb. 1958)
The action of CCl₄ on mono-, di-, and some tri-nitro
derivatives of benzene, benzenesulphonyl chloride,
naphthalene, diphenyl, phthalic acid or its anhydride,
phthalimide, and anthraquinone has been investigated and
the replacement of a nitro group by a chlorine atom has
been carried out with a 90% yield. As might be expected,
the labile bromine atom in bromonitrobenzenes is simultaneously replaced by chlorine; similarly, the SO₃Cl group
may be replaced in the corresponding derivatives. Resinification and carbonisation result when CCl₄ reacts with
nitrotoluenes, nitroanilines, and their benzoyl derivatives,
and with nitrophenols. This method, where applicable,
can form the basis of a quantitative estimation of the nitro
group. A mechanism for the replacement reaction, involving the formation of phosgene, is suggested. G.J.K.

Pentafluoroaniline E. J. Forbes, R. D. Richardson, and J. C. Tatlow

Chem. and Ind., 630–631 (24 May 1958) Pentafluoroaniline, m.p. $34^{\circ}v$., is the sole product obtained in high yield by the reaction of hexafluorobenzene with sodamide in liquid ammonia at $-70^{\circ}v$. Diszotisation of pentafluoroaniline under normal conditions gives the diazoamino compound. Solutions of pentafluorobenzene-diazonium salts may be obtained by working in non-aqueous media, e.g. acetic acid, or in concentrated aqueous mineral acid, e.g. $70^{\circ}w$ sulphuric acid. Under neutral or alkaline conditions the diazonium ion is unstable, and fluorine atoms are displaced from the nucleus; e.g. the deep red compound obtained when pentafluorobenzene-diazonium chloride is treated with alkaline β -naphthol contains both chlorine and hydroxyl groups in place of fluorine.

Monoazo Disperse Dyes containing a 3-Oxobutyl or 3-Hydrovybutyl Group

3-Hydroxybutyl Group N. Kuroki, T. Maruyama, and K. Konishi

Kôgyô Kagaku Zasshi, 59, 905-909 (1956):

Chem. Abs., 52, 8561 (25 May 1958)
disperse dyes were prepared by coupling N-

Monoazo disperse dyes were prepared by coupling N-ethyl-N-(3-oxobutyl)aniline and N-(2-hydroxyethyl)-N-(3-oxobutyl)aniline with p-nitroaniline, 2:4-dinitroaniline or 2:6-dichloro-4-nitroaniline. The dyeing properties of the products for acetate rayon, Vinylon and Amilan are recorded together with their absorption spectra and m.p. Those dyes containing a hydroxyethyl group had the better dyeing properties. Similarly monoazo dyes were prepared from the above nitroanilines and N-(3-hydroxybutyl)aniline and N-(2-hydroxyethyl)-N-(3-hydroxybutyl)aniline. These dyes had better dyeing properties than those containing oxobutyl groups. The difference in dyeing properties is discussed with reference to their structures. C.O.C.

Monoazo Disperse Dyes containing the Phenylmorpholine Ring

N. Kuroki, J. Nishino, and K. Korishi

Kôgyô, Kagaku Zasshi, 59, 909–912 (1956):

Chem. Abs., 52, 8561 (25 May 1958)

Monoazo dyes were prepared using as coupling

components 4-phenylmorpholine, 2-methyl-4-phenylmorpholine, 2:6-dimethyl-4-phenylmorpholine, 2-chloro-methyl-4-phenylmorpholine, 2-acetoxymethyl-4-phenyl-morpholine, and 2-hydroxymethyl-4-phenylmorpholine, and as diazo components p-nitroaniline, 2:4-dinitroaniline, and 2:6-dichloro-4-nitroaniline. The dyes containing acetoxymethyl and hydroxymethyl radicals had good affinity for acetate rayon, Vinylon and Amilan, the affinity of the others being poor.

Mechanism of the Coupling of Diazonium Salts with Heterocyclic Compounds. III—Indazole R. D. Brown, B. A. W. Coller, and M. L. Heffernan

J.C.S., 1776-1779 (May 1958) Variation in the rate of coupling of indazole with diazo-sulphanilic acid over the pH range 8-5-10-1 indicates that the reaction involves only the small proportion of anion in equilibrium with the neutral indazole mol. in these soln. This observed orientation in the coupling reaction is thus indicative of the relative reactivities of the various ring positions in the anion rather than in the free base. The preferential attack of the 3-position is in agreement with qual. molecular-orbital predictions on the assumption that both tertiary N atoms are less electronegative than the ring C atoms (cf. J.C.S., 1827 (1949)). Some kinetic evidence suggests that the azo cpd. formed initially undergoes a further reaction with another mol. of the diazonium cpd. H.H.H

Application of the Pariser and Parr Method to Dye

Ions with Amidinium Resonance S. P. McGlynn and W. T. Simpson

J. Chem. Phys., 28, 297-300 (1958):
Chem. Abs., 52, 8717 (10 June 1958)
The transition energies and transition probabilities for the transition energies and transition probabilities for the family of dye ions $(CH_3)_2N\cdot(CH:CH)_{g-1}\cdot CH:N^+(CH_3)_g$, were calculated by the Pariser and Parr method. The N's were taken into account by a perturbation method. Configuration interaction considering single excitations from the ground configuration was used. In the comparison with experimental results, however, it was considered better to use the diagonal matrix elements for the transition energies.

Orange to Red Azo Disperse Dyes from 1-Methyl-2-(p-aminophenyl)benziminazole

G. Cordella

Ricerca sci., 27, 3089-3090 (1957):
Chem. Abs., 52, 7705 (10 May 1958)
Synthesis of disperse dyes of formula 1-methyl-2-(p-ylazophenyl)benziminasole arylazophenyl)benziminazole $(aryl = C_aH_aN(CH_a)_3$ naphthol are not as fast to light as the others.

Cyanocarbon Chemistry

I-Preparation and Reactions of Tetracyano-

ethylene ethylene
T. L. Cairns, R. A. Carboni, D. D. Coffman, A. V. Engelhardt, R. E. Heckert, E. L. Little, E. G. McGeer, B. C. McKusick, W. J. Middleton, R. M. Scribner, C. W. Theobald, and H. E. Winberg
J. Amer. Chem. Soc., 80, 2775-2778 (5 June 1958)
An account of the synthesis of the first percyanoclefin, tetracyanocthylene. This compound can undergo various reactions including addition, replacement and excellention.

reactions including addition, replacement and cyclisation reactions, to give dyes, strong acids and heterocyclic compounds.

II—Spectroscopic Studies of the Molecular Complexes of Tetracyanoethylene R. E. Merrifield and W. D. Phillips

Ibid., 2778-2782 Complex-formation equilibrium constants and spectroscopic data for twenty-three n-complexes involving tetracyancethylene and various aromatic molecules are

III—Addition Reactions of Tetracyanoethylene W. J. Middleton, R. E. Heckert, E. L. Little, and C. G. Krespan

Ibid., 2783-2788 A study of reactions involving additions to the double bond of tetracyanoethylene. Tetracyanoethylene is an extremely active dienophile in reactions with conjugated Several unusual organic acids containing the

-C(CN)2C(CN)2H group were prepared from tetracyanoethylene. Acidie tetracyanoethane was formed reduction of tetracyanoethylene with H, thiols and HI, and acidic $\beta\beta\gamma\gamma$ -tetracyanoketones were formed by adding ketones to tetracyanoethylene. Sulphurous acid and tetracyanoethylene form 1:1:2:2-tetracyanosulphonic acid. Several salts of this dibasic acid are described.

IV— Dicyanoketene Acetals W. J. Middleton and V. A. Engelhardt

Ibid., 2788-2795 Dicyanoketene acetals are obtained by the urea-catalysed reaction of tetracyanoacetal with alcohols. They react with ammonia and primary and secondary amines to give 1-amino-1-alkoxy-2:2-dicyanoethylenes and 1:1-diamino-2:2-dicyanoethylene. The dicyanoketene cyclic acetals act as alkylating agents toward tertiary amines, alkylsulphides, thioureas, thioamides and thiosemicarbazide.

V- Cyanocarbon Acids and their Salts W. J. Middleton, E. L. Little, D. D. Coffman, and V. A. Engelhardt

Ibid., 2795-2806 An account of several new cyanocarbonacids whose acidic strengths rival those of the mineral acids. Several yield resonance-stabilised anions composed entirely of C and N.

VI—Tricyanovinylamines— a New Class of Dyes B. C. McKusick, R. E. Heckert, T. L. Cairns, D. D. Coffman, and H. F. Mower

Ibid., 2806-2815 Tetracyanoethylene reacts with primary and some secondary amines to yield N-tricyanovinylamines. It usually reacts with secondary and tertiary arylamine by attacking the aromatic ring to yield 4-tricyanovinylarylamines, e.g.

> (CH₃)₂N C(CN):C(CN)

These are a new class of dyes having strong affinity for hydrophobic fibres, e.g. polyester and polyacrylonitrile fibres. They are mostly brilliant reds and blues of good fastness to washing. Properties of 50 such dyes are given. The best yet prepared is 4-tricyanovinyl-N-methyl-N-(2-benzoxyethylaniline), $4 \cdot C_4H_5 \text{COOCH}_5 \text{CH}_4 \text{N}(\text{CH})_5 \cdot C_6H_4 \text{C}(\text{CN}) \cdot \text{C}(\text{CN})_2$ whose dyeings have good fastness to washing, sublimation and light. It can be dyed at pH 3-6 without serious decomposition but like other simple tricyanovinylarylamine dyes it rapidly decompos pH > 7, this decomposition involving replacement of the a-eyano group by hydroxyl. In an effort to produce dyes of other colours and better properties analogues of tricyanovinylarylamines were prepared. One analogue, prepared by condensing ethyl 4-dimethylaminoglyoxylate with malononitrile, had a carbethoxy group instead of a eyano group in the a-position of the vinyl group-

4-(CH₃)₂C₄H₄C(COOC₂H₅):C(CN)₂

The other analogues had a chromophore other than cyano at the β -position of the aryl group

4-R₃NC₆H₄C(CN):C(CN)Z

(Z = COAr, COOR, etc.); they were obtained by condensing a 4-aminobenzaldehyde with a compound having an active methylene group, adding HCN to the product and oxidising. The same method was used to prepare the deep blue dye 4-(4-dimethylaminophenyl)-1:3-butadiene-1:1:2-tri-carbonitrile. In alcoholic solution the colour is destroyed by addition of alkali but is restored on acidification, the ability of the alkaline solution to become blue on acidification is only gradually lost over a period of hours. This suggests that alkali reversibly converts the dye to a colourless intermediate (A), which gradually and irreversibly breaks down to compound B. Such intermediates are probably formed during the far faster basic hydrolysis of the 4-tricyanovinylarylamine-

 $4-(CH_2)_8NC_6H_4CH:CHC(CN):C(CN)_9 \xrightarrow{OH^-}_{H^+}$

4-(CH₂)₂NC₄H₄CH:CHC(CN)(OH)C⁻(CN)₃ ->

4-(CH₃)₈NC₆H₄CH:CHC(OH):C(CN)₂ + CN-

The analogues of 4-trievanovinvlarylamines share their virtues and defects as dyes. None prepared had dyeing properties as good as the best 4-tricyanovinylarylamines.

Thiazolecarbocyanines with Aryl Groupings in the Thiazole Nucleus

III— Methoxyarylthiazolecarbocyanines

Ukraimian Chem. J., 24, 79-88 (Feb. 1958)

New 5-methoxyaryl-2-methylthiazoles have been prepared, and their quaternary salts have yielded monoand tri-methins and also styrene derivatives. The absorption spectra of these dyes have been determined, and show that the electron-donating effect of the alkoxyl group is minimised when this substituent is separated from the main absorbing conjugated system by an aromatic nucleus.

IV - Steric Hindrance in 5-a-Naphthylthiazoles

2-Methyl-5-a-naphthylthiazole (I) and 2-methyl-5-a-(and β)-naphthyloxazole are synthesised, and the corres ponding cyanine and styryl dyes are obtained from the quaternary salts. The absorption spectrum of I in ethanolic solution shows that the molecule is sterically hindered and that the naphthalene nucleus does not lie in the plane of the thiszole ring.

V-Biscyanines and Bis-styryls of the Thiazole

The synthesis of 4:4'-bis-(2-methylthiazol-4 (and 5)-vl)diphenyl is given, and the corresponding "biscyanine" and "bis-styryl" dyes are obtained from the quaternary salts. Their absorption spectra in ethanolic solution are determined.

Trinuclear Sensitising Dves. VI-Isomers of Oxacvanine

V. Hishiki

Kagaku Kenyûsho Hôkoku, 30, 279 (1954):

Chem. Abs., 52, 8807 (10 June 1958) An account of the synthesis of the violet dye [2-(1-methylbenzoxazole)] [2'-(1'-ethylbenzoxazole)] [y-2''-(1''-methylbenzoxazole)] - pentamethincyanine 1-1'' -diiodide and the red dye [2-(1-methylbenzoxazole)] [y-2''-(1''-ethylbenzoxazole)] pentamethincyanine 1:1''-diiodide together with two other isomers. Spectrographic data of the four dyes are given. C.O.C.

Light Absorption of Condensed Aromatic Compounds. II-III A. Kiss

Acta Chimica Academiae Scientiarum Hungaricae,

11, (1-2), 85-111 (1957):

Hungarian Tech. Abs., 10, (1), 4 (1958) The theory of light absorption further developed by the author furnishes a satisfactory interpretation for the light absorption of two-axis acenes from among the linearly condensed aromatic compounds. Structural anomalies observed in the absorption of di- and tri-substituted naphthalene derivatives may be explained by substituents interfering with mesomerism. For angularly condensed aromatic compounds conclusions based on the improved theory agree in general with the experimental observations and data. With all compounds tested, bands obtained experimentally could be coordinated with the corresponding assumed excitation axes. The validity of band co-ordinations is confirmed by the absorption curves of mono derivatives. If the structural formulae of the compounds are known, the shape of the absorption curves can be CJWH established in advance.

Ultraviolet Absorption Spectra of Selenonaphthen, Selenoindigo, and 3-Hydroxyselenonaphthen A. I. Kiss and B. R. Muth

Acta Chimica Academiae Scientiarum Hungaricae,

11, (1-2), 57-61 (1957):

Hungarian Tech. Abs., 10, (1), 5 (1958) The absorption curve of 3-hydroxyselenonaphthen in 96% ethanol differs essentially from that of selenonaphthen. The spectrum of selenonaphthen consists of three regions of absorption, which indicates the presence of a condensed cyclic system. The curves of selenoindigo measured in three different solvents (chloroform, toluene, and dioxan) were characteristic of the indigo structure, consisting of two regions of absorption, one in the visible, the other in the ultraviolet region. C.J.W.H.

Dyeing of Wool with Lichens and Lichen Substances Y. J. Solberg

Acta Chem. Scand., 10, 1116-1123 (1956): Chem. Abs., 52, 7709 (10 May 1958)

Most non-Orseille lichens and lichen substances used for dveing wool contain at least one aromatic aldehyde group dyeing wool contain at least one aromatic antenyue group which with the free NH₂ groups in wool forms azomethin compounds which yield the bright yellow obtained under mild dyeing conditions. Under more drastic conditions, darker colours develop, indicating a more complex chemical process. chemical process. Furnaroprotocetrarie acid (I) was isolated from Cetraria islandica and salazinic acid (II) from Parmelia sazatilis, but the dyeing properties of Ramalina calicaro are attributable to quite another type of dye formation. The following azomethin compounds, all hygroscopic and decomposing at > 200°c, were synthesised scopic and decomposing at > 200 c, were synthesised the N-(p-aminobenzoic acid), the N-(g-glycine CH_3 ester), the N- $(CH_3$ anthranilate) and the N- $(CH_3$ m-aminobenzoate) of I and the NN-bis(CH₂m-aminobenzoate) of II.

Separation of Curcumin (C.I. 75300) from Curcuma Roots

A. D. Gol'tman

Ukrain. Khim. Zhur. 23, 659-661 (1957): Chem. Abs., 52, 8466 (25 May 1958)

The ground roots are treated with ethanol for 24 hr. and the mixture filtered. The residue is washed with ethanol. The curcumin is precipitated from the filtrate and wash liquor with satd. aq. Pb acetate, filtered off, washed with ethanol and water, suspended in water and treated with H_2S to decompose the Pb salt, filtered off, washed with water, dried at 80° C., dissolved in benzene, placed on a CaCO_a chromatographic column and cluted with 1% ethanol in benzene. The yield is 0.3%.

Red Vegetable Dye from Chicory

Naturwissenschaften, 45, 4041 (1958): Chem. Abs., 52, 8561 (25 May 1958)

A red dye isolated from the leaves of Cichorium intubus and C. endiva contains N and is insoluble in most solvents except water and alcohol. It is adsorbed quickly on tale and more slowly on cellulose. Spectrographic data are

Ommochromes. XII- Preparation of Pure Rhodommatin and Ommatin D to establish the Structure of Rhodommatin

A. Butenandt, E. Biekert, and R. Beckmann

Ann., 607, 207-214 (1957):

Chem. Abs., 52, 8145 (25 May 1958) Besides rhodommatin, a new red native ommatin dve. Ommatin D, has been identified by paper chromatography in fresh secretions of the butterfly Vanessa urricae. Ommatin D has a phenylquinonimine structure. Rhodommatin has empirical formula C20H16N2O10 and various hydrolyses etc. made in an effort to determine its structure are described.

Absorption Spectra of the Anthocyanin Pigment of Red Cabbage— a Natural Wide-range pH Indicator F. T. Wolf

Physiol. Plantarum, 9, 559–566 (1956): Chem. Abs., 52, 8832 (10 June 1958)

The purified pigment from Brassica oleracea changes over pH 1-10 from red to blue to green to yellow. This is caused by a drop in an absorption peak at 530 mµ. on changing pH from 1-7 accompanied by a rise in an absorption maximum at 615 mµ. in going from pH 3 to 8.
The latter peak drops in going to pH 10 while absorption at lower than 460 mµ, rises on increasing pH from 7 to 10.

Chlorophyll and Related Compounds VII - Structure of Bacteriochlorophyll J. H. Golden, R. P. Linstead, and C. H. Whitham *J.C.S.*, 1725–1732 (May 1958)

The red and the purple photosynthetic bacteria contain, in addition to carotenoids, one chlorophyll component, bacteriochlorophyll (1), which appears to be associated in the cell with different proteins. The chlorophyllic component of the green sulphur bacteria appears to be a different substance. Hans Fischer's structure for I is now strengthened by further data, and some aspects of its stereochemistry are elucidated. Stepwise dehydrogenation of a deriv. of I to the corresponding chlorin and porphin is described, together with some observations on the light absorption of the pigments.

ннн Carotenoid-sensitised Fluorescence of Chlorophyll in vitro

Nature, 181, 415-416 (8 Feb. 1958) Measurements of the fluorescence spectra of chromatographically purified chlorophyll A or B dispersed with β-carotene, lutein, or fucoxanthol in detergent micelles, show that energy absorbed by the carotenoid is transferred to chlorophyll with an efficiency comparable with that observed in chloroplasts. Transfer depends, inter alia, upon the proximity of the molecules participating and the over-lap between the spectra of the participants. Although the energy transfer from fucoxanthol to chlorophyll A is 100%, there is no evidence for the formation, in vitro, of a complex similar to that formed in the brown algae.

Carotenoid Syntheses O. Isler and M. Montavon

Chimia (Switz.), 12, 1-17 (1958): Chem. Abs., 52, 8093 (25 May 1958)

C.O.C.

of β- and α-Carotene with Bromosuccinimide

R. Entschel and P. Karrer Review, 25 references,

Purification of Carotene
B. G. Savinov and A. A. Svishchuk
Vitaminy, Akad. Nauk Ukr. S.S.R., 2, 17-21 (1956):
Chem. Abs., 52, 7617 (10 May 1958)

Carrot juice (ca. 80% water) is treated with solid NaOH. The saponified mixture is then treated with 4 times its volume of a chlorinated hydrocarbon. The solvent is evaporated and the residue mixed with silica gel. This mixture is placed in a column and fatty impurities washed out with solvents which do not dissolve carotene. Then the sterols are removed by washing with boiling CH₃OH. Finally the pure carotene is dissolved in chlorinated hydrocarbon and recrystallised.

Some Cleavage Products of the Boron Trifluoride Complexes of α-Carotene (C.I. 75130), Lycopene (C.I. 75125) and γ-Carotene (C.I. 75130) W. V. Bush and L. Zechmeister

J. Amer. Chem. Soc., 80, 2991-2999 (20 June 1958) Hydrolysis of the deeply coloured a-carotene-BF, complex yields mainly 4-hydroxy-a-carotene while the a-ionone end of the molecule remains unaltered. Dea-ionone end of the molecule remains unaltered. Dehydrogenation of the hydroxy compound yields 4-keto-a-carotene. Ethanolysis of the complex yields 4-ethoxy-a-carotene. Similarly treated, the BF₃ complexes of lycopene and γ-carotene yielded 5:6-dihydroxy-5:-6-dihydrolycopene and 4-hydroxy-γ-carotene respectively. The cyclic terminal group of the γ-carotene complex seems to be more reactive than its acyclic end. C.O.C.

Preparation of Stable Yellow Ferric Pigments M. Kranz

Przemyal Chem., 13, 646-653 (1957):

Chem. Abs., 52, 9625 (10 June 1958) Bright, full yellow pigments are obtained from ferrie isoorthohydroxide or its sulphate by ageing them in aq. NaOH, filtering and drying at room temperature. The NaOH should contain a very small amount of Na silicate. The pigments obtained are composed of a-Fe0OH or a-Fe₂O₃·H₂O. FeSO₄·7H₂O (1 kg.) was dissolved in distilled water (6 l.), filtered, and 3% H_2O_2 (3 l.) added and the mixture stood for 12 hr. The ppt. was then filtered (Buchner) and washed until free of Fe⁺⁺⁺ ions. The yellow precipitate was then dumped into 0.5 x NaOH (31.) containing Na₂SO₄·10H₂O (6·6 g.) and there aged for 5-7 days at room temperature. The precipitate was then filtered off (Buchner), washed until free of OH⁻, dried at room temperature and ground. The product had sp.gr. 3.56, bulked 0.35, had total water content 14.77% (lost 2.62% at 110°c.), becomes dark at 160° and brick red at > 200°c. By heating for a short time at 860-880°c. an intense red was obtained. The yellow pigment met the requirements for high-class inorganic pigments. C.O.C.

Cadmium Vermilion, a New Class of Pigments H. Pfleiderer

Fette, Seifen, Anstrichmittel, 58, 1073-1076 (1956) Preparation, applications and properties of the Cadmium Vermilion pigments (G8) are described. They are a mixed-crystal type composed of Cd and Hg sulphides, a probable structure being postulated from consideration of X-ray absorption. C.O.C.

Production of Lead Cyanamide (C.I. Pigment Yellow 48) and its Use as a Varnish and Paint Pigment V. G. Brudz, R. L. Globus, L. I. Gracheva, and A. M.

Grozovskava Khim, Prom., 352-356 (1957):

Chem. Abs., 52, 9626 (10 June 1958) PbNCN contains less Pb than litharge (C.I. Pigment Yellow 46), but all the Pb takes part in the interaction with oils as against only 66% of Pb in minium (C.I. Pigment Red 105). PbNCN paints are stable, have high covering power and are easy to use. Production methods for PbNCN are reviewed and discussed. C.O.C.

Lead Cyanamide (C.I. Pigment Yellow 48)

 $Przemysl~Chem.,~13,~688-690~(1957):\\ Chem.~Abs.,~52,~9625~(10~Juno~1958)\\ CaNCN~(100~g.)~was slowly added to 7.5-8%~H_28O_4~(11.),\\ the mixture was vigorously agitated and cooled to <math><45^{\circ}$ C. The final mixture was only slightly acidic. The CaSO4 was filtered off and aq. H₂SO₄ added to the filtrate to prevent hydrolysis with consequent precipitation. This was followed by addition of aq. lead nitrate (5-10 ml.) to remove H₂S. The PbS was filtered off, and Pb nitrate (600 ml. containing 160-165 g.) added. Only slight precipitation occurred. Milk of lime was added to bring the pH to slightly above 8. A large precipitate was formed which changed from light yellow to a vivid yellow with a greenish tint. The pigment was filtered off and dried at 70-80°c. The yield was 125 g. and contained 82% Pb. C.O.C.

Yellow Monoazo Disperse Dyes for Polyester Fibres

Diazotised aniline derivatives containing 1 or 2 carboxylie ester or carboxylamide groups are coupled with 3methyl-1-phenyl-5-pyrazolones free of COOH and SO4H groups to give yellow monoago dyes of good fastness to light and sublimation on polyester fibres. Thus methyl p-aminobenzoate is diazotised and coupled with 3-methyl-1-phenyl-5-pyrazolone to give-

E.8.

Metal(Chromium and Cobalt)-complex Monoazo Dyes for Wool, Nylon, etc.

The 1:2-Co and Cr complexes of inonoazo dyes free from COOH and SO₂H groups obtained by coupling a diazotised o-aminophenol with 1-ureido-7-naphthol (I) dye wool, nylon etc. from neutral or weakly acid or weakly alkaline baths. I is obtained from 1-amino-7-naphthol and potassium cyanate. Coupling with diazotised 2-amino-4-chlorophenol for instance gives—

converted by boiling with aq. sodium chromosalicylate into the Cr complex which is a grey.

Metal(Chromium and Cobalt)-complex Monoazo Wool Dyes having a Urea Residue

FBy
Diazotised o-aminophenols free of SO₄H and COOH
groups are coupled with o-coupling naphthols or keto-enol
compounds having a urea residue -NH-CO-NH₂, and the
products converted into 1:2-metal:dye complexes which
dye wool, nylon, etc. from neutral or weakly acid baths.
Thus 2-amino-4-ethylsulphonylphenol is diazotised and
coupled with 2-naphthol-8-urea (from 8-amino-2-naphthol
and KCNO) to give—

This is dissolved in aq. NaOH and boiled with $K_4Cr_4O_7$ and glucose to give the Cr-complex which dyes wool blue-grey. Fig.

Brown and Green Metal(Chromium and Cobalt)complex Monoazo Wool Dyes from 2-Aryl-5-aminobenztriazoles

The Cr and Co complexes of monoazo compounds free of COOH and SO₂H groups obtained by coupling diazotised c-aminophenols with 2-aryl-5-aminobenztriazoles dye wool, nylon, etc. from neutral or weakly acid baths. Thus 2-aminophenol-4-sulphonamide is diazotised and the suspension so formed added to a dispersion in water of 2-phenyl-5-aminobenztriazole. The monoazo compound formed is dissolved in hot aq. NaOH and aq. ammoniacal cobalt chloride added to give the Co complex of the o-amino-o'-hydroxymonoazo compound—

which dves wool dark brown.

E.S.

Yellow and Brown Metal(Cobalt)-complex Monoazo Wool Dyes Derived from Arylides of β-Aminocrotonic Acid

The Co complexes of monoazo dyes obtained by coupling a diazotised σ-aminophenolsulphonamide with an arylide of β-aminocrotonic acid dye wool yellow or brown from neutral baths. Thus acetoacet-σ-anisidide is stirred with aq. NH₃ at 70°c. to give—

This is coupled with diazotised 2-aminophenol-4-sulphon-anilide to give the monoazo compound—

$$\begin{array}{c} \text{OH} \ \text{H}_2\text{N} \cdot \text{C} \\ \text{OH} \ \text{H}_2\text{N} \cdot \text{C} \\ \text{C}_6\text{H}_6 \cdot \text{NH} \cdot \text{SO}_3 \end{array} \quad \begin{array}{c} \text{CH}_3\text{O} \\ \text{CH}_3\text{O} \end{array}$$

which is dissolved in aq. NaOH and stirred at 70–80°c. with aq. CoSO₄ to give the Co complex, which dyes wool yellow brown.

Red, Metal(Chromium)-complex Monoazo Pyrazolone Dyes for Wool, Nylon, etc.

BP 796,381

An addition to BP 754,270 (J.S.D.C., 72, 544 (1956)). The same diazo component, viz. 2-amino-4-chloro(or methyl)-6-nitrophenol, is diazotised and coupled with pyrazolones—

(R = a butyl or amyl radical substituted by one or more OH groups) and the products converted into their 1:2 Cr complexes which dye wool, nylon, etc. red from neutral or weakly acid baths. Thus 2-amino-4-chloro-6-nitrophenol is diazotised and coupled with 1-phenyl-3-methyl-5-pyrazolone-3'-sulphon(\(\beta\)-methylolpropylamide, and the product heated with ammonium chromium sulphate in formamide to give the Cr complex, which is a bluish red.

Azo Vat Dyes of the Triazine Anthraquinone Series American Cyanamid Co. USP 2,800,468

Vat dyes of general formula—

(Aq = an anthraquinonyl residue; R = Ar of benzene series in which the azo group is in other than o-position to the triazine ring) are yellows to reds, the azo groups being unaffected by normal vatting conditions. A variety of syntheses are described, the preferred method starting with e.g. a benzoguanamide, which is first nitrated and then treated as follows—

Condensation with 4 mol. of α -aminoanthraquinone, with replacement of each Cl by an anthraquinonylamino group, gives a yellow vat dye. E.S.

Green and Olive Metal(Copper)-complex Trisazo Direct Dyes

FBy BP 796,397
The title dyes are the Cu complexes of trisazo dyes of general formula—

(X = H or SO₂H; R-OH = residue of an aminonaphtholmono- or di-sulphonic acid, OH being orthe to the azo link). Thus the trisage compound 5-sulpheanthranilic acid 1-1p-aminophenyl-3-methyl-5-pyrazolone $\xrightarrow{2}$ 2-ethoxy-1naphthylamine-6-sulphonic acid 3-N-phenyl-J acid is boiled with aq. CuSO4 to give a green.

Anthraquinone Disperse Dyes

BP 797,383

Reddish-blue dyes for cellulose acetate and synthetic polyester fibres which have outstanding fastness to light and dry heat treatments are of formula

(R = H, CH₃, OCH₃; X = a direct link or the $-C_nH_{2n}CO_3$ group; Y = a direct link or the $-(C_m H_{2m}O_p)_p$ group; Z = a direct link or the $-(C_q H_{2q})_p$ group; n, q = 1-4; m = 2-3; p = 1-3; X, Y and Z do not all simultaneously stand for a direct link). They are made from 2-bromo-4anilino-1-hydroxyanthraquinone by (a) reacting with Na polysulphide to form the 2-mercaptan; (b) treating the Na derivative of the mercaptan with H-Z-Y-X-Hal e.g. butyl bromide, β-butoxyethyl bromide, hydroxyethylchloro-acetate, etc. to give-

and (c) oxidising this product with H₂O₂ or MnO₂ by heating in a suitable solvent e.g. glacial acetic acid. ET

Halogenosalicyloylamidoanthraquinone Pigments

American Cyanamid Co.

BP 795,919 1-Salicyloylamidoanthraquinone is well known as a greenish-yellow pigment particularly suitable for mixing with blue pigments to produce bright greens. A serious weakness is its poor bleed resistance which is due to its solubility in the solvents used in formulating enamels and lacquers. It is now found that excellent bleed resistance and improved light fastness can be achieved if a halogen substituent is present in the 3-position of the salicyloyl ring. These pigments are made by condensing 1-aminoanthraquinone with the acid chlorides of halosalicylic acid. Thus, 1-aminoanthraquinone (22·3) and 3:5-dichlorosalicylic acid in nitrobenzene (300) are heated at 105°c.
Thionylchloride (15.5) in nitrobenzene (24) is added gradually at 105-110°c. Acylation is completed at 125-130°c. The product crystallises on cooling and is separated by filtration and washing. Acid pasting and re-precipitation precede the conversion of the pigment to the form in which it is applied.

Yellow Quinophthalone Dyes for Hydrophobic Fibres

Quinophthalone compounds of formula-

(R = alkylene radical of 2-10 C; the group -NR¹R³ = nitrogenous base radical, e.g. dialkylamine, monoalkylamine, radicalkylamine, radicalk monohydroxyalkylamine, dihydroxyalkylamine, piperidine, morpholine; X and Y = H, Cl, Br or CH₂) are themselves yellow disperse dyes for nylon, Terylene, cellulose acetate or polyacrylonitrile fibres and are also intermediates for cationic (basic) dyes for nylon or polyacrylonitrile from acid, neutral or alkaline baths. intermediate disperse dyes are made by reacting

(R = a 3-hydroxyquinolyl radical) with a monotortiary $\left(\mathbf{H_{8}NRN_{R^{3}}^{/R^{1}}} \right)$ (alkylene of 2-10 C). alkylene diamine

Reaction occurs in a heated inert solvent, e.g. nitrobenzene, in presence of an organic acid-binding agent, e.g. Na acetate. Conversion to basic dyes is carried out by reacting the intermediates with quaternising agents, e.g. dimethyl sulphate, triethyl phosphate, ethyl bromide, etc., the resulting compounds being then isolated as water-soluble ZnCl, double salts.

"Benzanthronimide Green" Vat Dyes

BP 797,240 The olive-green vat dyes made by the method of BP 773,116 (J.S.D.C., 73, 405 (1957)) are made greener by after-treating with acid condensing agents, e.g. solutions of AlCl₃ in thionyl-, sulphuryl-, benzene sulpho-, benzoyl-, oxalyl-, methane sulpho- or chloromethane sulpho- ohlorides. Thus, the dye (6·7 g.) obtained by treating Bz-1-benzanthronyl-5-amino-1:1'-dianthrimide with AlCl₃ and pyridine is added to benzenesulphochloride (60 ml.) and AlCla (30 g.) and heated 6 hr. at 60°c. The melt is poured into dil. NaOH solution, boiled, filtered and the residue is washed neutral.

Copper Phthalocyanine Precursor DuP

USP 2,795,586

This product, which gives Cu phthalocyanine by reaction with reducing agent, fias the formula-

$$\begin{array}{c|cccc} & & & & & & & & & \\ & & & & & & & & \\ & & & & & & & \\ & & & & & & & \\ & & & & & & \\ & & & & & & \\ & & & & & \\ & & & & & \\ & & & & & \\ & & & & \\ & & & & \\ & & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & & \\ & & \\ & & & \\ & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ & & \\ &$$

and is prepared by heating phthalonitrile, an anhydr. cupric salt and ammonia at > 90°c. in an alcoholic solvent containing a catalyst, e.g. urea, USP 2,772,284. method and the similar methods of USP 2,772,283 and 2,772,285 the final product is recovered by drowning into water, filtering and drying. It is stated that products separated in this way, which are amorphous, hydrolyse readily in contact with moisture and especially during drying. The present invention claims that this instability can be overcome by slurrying the final water-wet filtercake with a low-boiling water-miscible alcohol, e.g. methyl, othyl or isopropyl, and then drying at $< 60^{\circ}$ c. The product is then crystalline, has higher initial purity and good storage stability.

Production of Copper Phthalocyanine Precursor DuP

In earlier patents (viz. USP 2,772,283 and 2,772,284) reaction between phthalonitrile, cupric salt and NH, was carried out in a neutral organic solvent, e.g. methanol, ethanol, Cellosolve, dimethyl formamide, etc. The present patent claims that this list of solvents can be extended by inclusion of organic sulphoxide compounds which give a inal product in excellent yield and of high purity. Thus, NH₄ is passed into dimethyl sulphoxide (138) and CuSO₄,H₃O (20-5) for 2 hr. at 25-40°c. Mixed phthalonitrile (64) and methylglucamine (4) are added and the mass is stirred at 85-80°c. for 1-2 hr. whilst passing a slow stream of air (6.5 parts by wt. of air during reaction seried). Filtratic represents the companies of the companies period). Filtration removes any Cu phthalocyanine formed and the precursor is separated by drowning the filtrates into water filtering and washing. The precursor may be

used as a dye for textiles by applying from an alcoholic or aq./alcoholic bath and developing by heat or reduction.

Production of Copper Phthalocyanine Precursor DuP

Cu phthalocyanine precursor in excellent yield and purity is made from 1:3-diimino-isoindoline (12), anhydr. CuCl₂ (1·8), dimethyl sulphoxide (70) by heating at 75°c. for 2 hr. then at 91°c. for 1.5 hr.

Hydrocarbon-soluble Phthalocyanines

USP 2,795,584 General Aniline Greenish-blue to greenish-yellow phthalocyanines having improved solubility in hydrocarbon solvents and useful for colouring printing inks and polystyrene are

$$\operatorname{Pey}_{-}^{\Gamma}(D)q\operatorname{-Ar}_{-}^{\Gamma}\left(\operatorname{CH}_{2}\operatorname{N}_{-}^{\operatorname{R}^{1}}\right)_{a}$$

Thus, e.g.

$$\operatorname{Pey} \left\{ \operatorname{SO}_2 \middle CH_3 \atop \operatorname{CH}_2 \operatorname{N}(\operatorname{decyl})_3 \right]$$

(Pcy = phthalocyanine nucleus; D = divalent bridging link; q = 0-1; $Ar = pendant aryl nucleus of <math>\gg 10$ C; $R^1 = H$, Alk, aralkyl, aryl, cycloalk or heterocyclic radical; $R^2 = Alk$, aralkyl, aryl, cycloalk or heterocyclic radical of $\ll 4$ C; R^1 , R^2 have no water-solubilising substituents and may together form a ring; n = 1-2; p = 1-8). They are made by reacting chloromethylated phthalocyanines with either (a) a large excess of a primary or sec. amine or (b) the amount of amine required by the -CH₂Cl groups present in an inert solvent which also contains an acid-binding agent, e.g. alkali metal carbonate.

Phthalocyanine Derivatives General Aniline USP 2,795,583 Greenish-blue to greenish-yellow phthalocyanines, of high light fustness, useful for colouring urea-HCHO resins, cellulose nitrate lacquers, spirit varnishes, etc. are produced

from phthalocyanines containing chloromethyl groups by

Pey
$$\left[A\right]_{m}$$
 $\left[CH_{2}CI\right]_{n}$ + n $\left[CH_{2}CI\right]_{n}$ OH $\left[CH_{2}CI\right]_{n}$ OH $\left[CH_{2}CI\right]_{n}$ OH $\left[CH_{2}CI\right]_{n}$ OH $\left[CH_{2}CI\right]_{n}$ + n HCl

group of formula (Pcy = phthalocyanine nucleus; A = (Fey = parameter) in the fraction of the first property of the first pendant aryl nucleus of \Rightarrow 10 cyclic C; m = 0–8; n < 1; R¹ = H, lower alk. or alkoxy). Examples of D in the above formula are -S-, -O-, -CO-, -CH₂O-, -SO₂O- etc. The chloromethyl phthalocyanines are made by condensing appropriate phthalocyanines at elevated temperature in presence of AlCl₃ and a tertiary amine with sym, bichloromethylether or paraformaldehyde (ref. USP 2,435,307; 2,542,327 and 2,542,328). Condensation of the intermediate with a β -resorcylic acid is carried out in an inert solvent, e.g. naphthalene, nitrobenzene, dichlorobenzene at 100-170°c. in presence of a catalyst, e.g. SnCl4, FeCl3, AlCl3, ZnCl₂, HgCl₂ etc. After reaction the product may be precipitated and collected by filtration. These colouring matters may also be used for alkali soluble inks and for colouring some fabrics where fastness to solvent cleaning is desirable. E.T.

Non-flocculating Phthalocyanine Pigments

USP 2,799,594 Pigments highly resistant to flocculation in organic coating compositions are obtained by use in the phthalicanhydride so that > 15% of the resulting phthalocyanine molecules contain the polar sulphonic group. Their tinetorial properties differ only very slightly from those of unmodified phthalocyanines and have the same excellent fastness to light and resistance to chemical attack.

USP 2,799,595

The above pigments while suspended in a chlorinated hydrocarbon are ground in a ball mill containing balls > 0.25 in. diameter and the liquid removed to yield a dry pigment of soft texture. C.O.C.

Carbon Black (C.I. Pigment Black 6) Houdry Process Corpn. USP 2,800,395 An improved method of obtaining Carbon Black by decomposing light hydrocarbons in presence of a catalyst. COC

Recovering Finely Divided Pigments from Streams

Godfrey L. Cabot USP 2,799,358 A method of recovering finely divided pigments in a dry state from aerosols flowing under pressure, e.g. of Carbon Black (C.I. Pigment Black 6) produced in high pressure

Cyanine Dyes containing Pyrazole Nuclei

BP 797,144 Hord Dyes of formula-

(R¹ = Ar which may be substituted by Hal, alkoxy or acylamino; R² and R³ = H, Alk or Ar; R⁴ = Alk; X = acid residue; D¹ = residue of a 5- or 6 membered nucleus; D² = residue of a 5- or 6-membered cyclicketomethylene compound) are produced by reacting a compound of formula-

with a compound containing a reactive methylene group. Thus the yellow 3-ethyl-2-[2'-(1"-phenyl-4"-pyrazolyl)vinyl]benzothiazolium p-toluene sulphonate is obtained by fusing 2-methylbenzothiazole with ethyl p-toluene sulphonate and then boiling it under reflux with 1-phenylpyrazole-4-aldehyde, ethanol and anhydrous Na acetate. C.O.C.

Fluorescent Brightening Agents

BP 796,211

(M = cation; n = 0, 1 or 2; X = acyl radical of a carbocyclic aromatic carboxylic acid) are prepared from compounds of formula-

by either (a) diazotising and then coupling with a naphthylamine in o-position to the amino group or with a mono- or di-sulphonic acid of such a naphthylamine, oxidising the resulting dye to the corresponding 1:2:3-triazol compound, reducing the NO, to NH₂ and acylating it with a compound containing the X radical, or (b) acylating the amino group, reducing the NO₂ to amino, diazotising and coupling in o-position to it with a naphthylamine or a mono- or disulphonic acid of a naphthylamine and finally oxidising the dye into the corresponding 1:2:3-triazol compound. Thus phenoxyacetyl chloride in chlorobenzene is dropped over $2 \text{ hr. at } 0-5^{\circ}\text{c.}$ into Na-4-(6'-sulphonaphtho-1',2:4:5triazol-(2))-4'-aminostilbene 2:2'-disulphonate in aqueous

sodium carbonate. The mixture is stirred for 16 hr., the chlorobenzene distilled off azeotropically with water at 100°c. after which water is added to precipitate Na-4-(6'-sulphonaphtho-1',2':4,5-triazolyl-(2)-4'-phenoxyacetylaminostilbene-2:2'-disulphonate. This has greenish yellow fluorescence in ultraviolet radiation and has good affinity for cellulosic and nitrogenous fibres. On fibres it shows strong bluish-green fluorescence which itself has no significant brightening effect but which can be used to correct the unwanted reddish tinge often obtained by use of fluorescent brightening agents. C.O.C.

Furnace Black (C.I. Pigment Black 7)

Godfrey L. Cabot

USP 2,801,157

A process and apparatus for producing Carbon Black from an oil "make" material combined with a gas auxiliary

Silica (C.I. Pigment White 27)

British Titan Products Co BP 796,735 Vaporised silicon tetrachloride is oxidised in a fluidised bed of inert material of mean particle size 40-1000 µ, and maintained at 500-1200°c. This yields fairly coarse flocks of silica gel, which are fluidised with air containing steam or with superheated steam, both at $> 250^{\circ}$ c., to yield a product whose aqueous suspension has pH 4–7. C.O.C.

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USP 2,798,819 An improved method of obtaining pigmentary TiO₂ by see vapour phase oxidation of TiCl₄. C.O.C. the vapour phase oxidation of TiCl4.

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Pelleting Finely Divided Solids such as Carbon Black with Liquids Godfrey L. Cabot

BP 795,263

Organic Silicate Compounds as Driers for Paints, etc. (III p. 660)

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Dyeing Properties of Azo Acid Dyes- I (VIII p. 669) Principles of Dyeing with Reactive Dyes (VIII p. 669)

Chemical Modification of Fibres and Dyes (VIII p. 669) Dyeing Nitrogenous Fibrous Materials with Anthraquinonoid Acid Dyes (VIII p. 670)

Colour Couplers (IX p. 671)

V-PAINTS; ENAMELS; INKS

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An account of work on several grades of mica, dry ground, wet ground and micronised. Tables and microphotographs indicate proportions of ingredients, adhesion of films and effects of wet grinding. In general, micronised mica yields better results than a normal dry ground type while the films are fresh but as they age the advantages are

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Off. Dig. Fed. Paint Varn. Prod. Cl., 30, 364-430 (April 1958)

A group of seven papers by various authors.

C.J.W.H.

PATENTS

Printing Inks containing Anti-skinning Agents International Minerals & Chemical Corpn.

USP 2.798,000

Incorporation of a little of an amino acid containing solution, e.g. a proteinaceous hydrolysate, into a printing ink renders the ink quick drying while remaining soft and pliable on the rollers. C.O.C.

Paints, Lacquers and like Coating Compositions N.V. Verf- en Vernis-fabriek W. Paulussen BP 79

A mixture of γ-benzene hexachloride (10-50 parts by wt.) and dichlorodiphenyltrichloroethane (90-50) is plastic and does not tend to crystallise. When incorporated in paints, etc. it tends to migrate to the upper surface of coatings formed by the paint. This gives the coatings a contact-insecticidal activity of long duration. C.O.C.

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National Starch Products Replacement of polyvinyl acetate as the principal resin base by aqueous emulsion of a copolymer of vinyl acetate and another monomer, e.g. dibutylfumarate, which yields a copolymer more plastic than vinyl acetate alone, enables emulsion paints to be made without use of a special plasticising agent.

Phosphorus-containing Polyester Resins for making Flame-resistant Paints (III p. 660)
Production of Lead Cyanamide (C.I. Pigment Yellow 48)

and its Use as a Varnish and Paint Pigment (IV p. 663)

VI—FIBRES; YARNS; FABRICS

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Maturity of Cotton Fibre for Cotton Spinning IV-Relations between the Maturity of Cotton Fibre and its Chemical Constituents

Y. Ono

J. Soc. Textile Cellulose Ind. Japan, 13, 785-791 (Dec. 1957)

Chemical analysis of cellulose and non-cellulose substances (nitrogen, phosphorus, ash, wax, and pectate) is related to maturity determined by physical methods.

V-Relations between the Maturity of Cotton Fibre and its Content of Reducing Sugars

Y. One and K. Sato

The contents of reducing and total sugars are related to maturity and to fineness- (1) The reducing-sugar content of the very immature lint is > 30%, but that of mature cotton fibre is 0.1% (oven-dry basis), and the reducing-sugar content of lint rapidly decreases as it matures. (2) The reducing-sugar contents of immature lint dyed green by the differential dyeing method vary between about 7 and 33%, and are extremely high compared with those of mature limit. These reducing sugars in immature lint comprise glucose, fructose, and a pentose. C.J.W.H.

Applications of Infrared Absorption Spectroscopy to Investigations of Cotton and Modified Cottons. I—Physical and Crystalline Modifications and Oxidation R. T. O'Conner, E. F. DuPré, and D. Mitcham Text. Research J., 28, 382–392 (May 1958) The infrared absorption spectra of cotton cellulose obtained by means of the KBr technique can be used to investigate changes in physical and crystalline properties. Experiments are described illustrating how the technique can be exployed to follow changes in hydrogen bending or can be employed to follow changes in hydrogen bonding or to measure the degree of crystallinity. Oxidation of cotton can be detected and measured by means of infrared absorption spectroscopy only if carbonyl, not mixtures of carbonyl and carboxyl, is produced by the oxidation

Relations between Supermolecular Structure and Mechanical Behaviour of Native and Chemically **Modified Cotton Cellulose**

V. W. Tripp, R. S. Orr, H. M. Ziifle, and C. M. Conrad Text. Research J., 28, 404-417 (May 1958) Cotton fibres are principally constructed of macromolecules of cellulose in various microfibrillate arrangements, which may collectively be called the supermolecular used as a dye for textiles by applying from an alcoholic or aq./alcoholic bath and developing by heat or reduction.

Production of Copper Phthalocyanine Precursor

Cu phthalocyanine precursor in excellent yield and purity is made from 1:3-diimino-isoindoline (12), anhydr. CuCl₂ (1.8), dimethyl sulphoxide (70) by heating at 75°c. for 2 hr. then at 91°c. for 1.5 hr.

Hydrocarbon-soluble Phthalocyanines

General Aniline USP 2,795,584 Greenish-blue to greenish-yellow phthalocyanines having improved solubility in hydrocarbon solvents and useful for colouring printing inks and polystyrene are-

$$\operatorname{Pey}_{-}^{\Gamma}(D)q - \operatorname{Ar}_{-}^{\Gamma}\left(\operatorname{CH}_{2}\operatorname{N}_{\operatorname{R}^{2}}^{\operatorname{R}^{1}}\right)_{n}$$

Thus, e.g.

$$\operatorname{Pey} \left[\operatorname{SO}_{3} \left(\operatorname{CH}_{3} \operatorname{N}(\operatorname{decyl})_{2} \right) \right]$$

(Pcy = phthalocyanine nucleus; D = divalent bridging link; q = 0-1; Ar = pendant aryl nucleus of $\gg 10$ C; R¹ = H, Alk, aralkyl, aryl, cycloalk or heterocyclic radical; R² = Alk, aralkyl, aryl, cycloalk or heterocyclic radical of $\ll 4$ C; R¹, R² have no water-solubilising substituents and may together form a ring; n = 1-2; p = 1-8). They are made by reacting chloromethylated phthalooyanines with either (a) a large excess of a primary or sec. amine or (b) the amount of amine required by the -CH₂Cl groups present in an inert solvent which also contains an acid-binding agent, e.g. alkali metal carbonate.

Phthalocyanine Derivatives

General Aniline Greenish-blue to greenish-yellow phthalocyanines, of high light fastness, useful for colouring urea-HCHO resins,

cellulose nitrate lacquers, spirit varnishes, etc. are produced from phthalocyanines containing chloromethyl groups by

Pey
$$\left[A\right]_{M}$$
 $\left[CH_{4}\right]_{COOH}$ $+ n$ HCl

(Pcy = phthalocyanine nucleus; A = group of formula –(D) –Ar–; D = divalent bridging link; q=0–1; Ar = pendant aryl nucleus of \Rightarrow 10 cyclic C; m=0–8; $n \leqslant 1$; R¹ = H, lower alk. or alkoxy). Examples of D in the above formula are –S-, –O-, –CO-, –CH₂O-, –SO₂O- etc. The chloromethyl phthalocyanines are made by condensing appropriate phthalocyaninos at elevated temperature in presence of AlCl₂ and a tertiary amine with sym. bichloromethylether or paraformaldehyde (ref. USP 2,435,307; 2,542,327 and 2,542,328). Condensation of the intermediate with a β -resorcylic acid is carried out in an inert solvent, e.g. naphthalene, nitrobenzene, dichlorobenzene at 100-170°c. in presence of a catalyst, e.g. SnCl₄, FeCl₃, AlCl₃, ZnCl₃, HgCl₂ etc. After reaction the product may be precipitated and collected by filtration. These colouring precipitated and collected by filtration. These colouring matters may also be used for alkali soluble inks and for colouring some fabrics where fastness to solvent cleaning is desirable.

Non-flocculating Phthalocyanine Pigments

DuP USP 2,799,594 Pigments highly resistant to flocculation in organic coating compositions are obtained by use in the phthalic-anhydride-urea synthesis of 4-sulphophthalic acid or anhydride so that > 15% of the resulting phthalocyanine molecules contain the polar sulphonic group. Their tinctorial properties differ only very slightly from those of unmodified phthalocyanines and have the same excellent fastness to light and resistance to chemical attack.

USP 2,799,595

The above pigments while suspended in a chlorinated hydrocarbon are ground in a ball mill containing balls ⇒ 0.25 in. diameter and the liquid removed to yield a dry pigment of soft texture.

Carbon Black (C.I. Pigment Black 6)

USP 2,800,395 Houdry Process Corpn.

An improved method of obtaining Carbon Black by decomposing light hydrocarbons in presence of a catalyst.

Recovering Finely Divided Pigments from Streams of Gases

Godfrey L. Cabot A method of recovering finely divided pigments in a dry state from aerosols flowing under pressure, e.g. of Carbon Black (C.I. Pigment Black 6) produced in high pressure

Cyanine Dyes containing Pyrazole Nuclei BP 797,144

(R1 = Ar which may be substituted by Hal, alkoxy or acylamino; R² and R³ = H, Alk or Ar; R⁴ = Alk; X = acid residue; D1 = residue of a 5- or 6, membered nucleus; D² = residue of a 5- or 6-membered cyclicketomethylene compound) are produced by reacting a compound of

with a compound containing a reactive methylene group. Thus the yellow 3-ethyl-2-[2'-(1''-phenyl-4''-pyrazolyl)-vinyl]benzothiazolium p-toluene sulphonate is obtained by fusing 2-methylbenzothiazole with ethyl p-toluene sulphonate is obtained by onate and then boiling it under reflux with 1-phenyl-pyrazole-4-aldehyde, ethanol and anhydrous Na acetate. C.O.C.

Fluorescent Brightening Agents

BP 796,211

Compounds of formula-CH:CH NHX SO₂M MO₂S (MOaS)a+

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BP 795,263

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Ibid., 791-796

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Cotton fibres are principally constructed of macromolecules of cellulose in various microfibrillate arrangements, which may collectively be called the supermolecular structure. It includes such features as crystallinity, D.P., and hydrogen bonding. Together with the macroscopic form of the fibre, many of these aspects have an influence on the response of the fibre to mechanical forces. Selected examples are presented, showing how the mechanical behaviour is conditioned in various ways by the supermolecular structure.

Structure of a-Keratin

M. L. Huggins

Proc. Natl. Acad. Sci. U.S., 43, 204-209 (1957):

Chem. Abs., 52, 8674 (10 June 1958)

A suggested new model for a-keratin has close packing of (right-handed) polypeptide helices, with a distance between helix axes of about 9 A. The helices are considered to be grouped into triplets, with the residue sequence patterns in each polypeptide helix shifted and rotated, relative to its two neighbours in the same triplet, so that equivalent residues are placed around a threefold screw axis, the triplet axis. The unit structure contains 81 chains in three equivalent groups of 27 chains. For an assumed unit distance of 95a. (to give the experimental d. of 1·32 g./o.c., if the average residue weight was 114 and the average translation per residue was 1·49a.), the calculated distance for forms 10·0, 20·0, 30·0, 63·0, and 90·0 are 82, 41, 27·4, 10.4 and 9.1a. respectively. The chains are adjacent to one another, in the same 27-chain group, in which corresponding residues are at the same height. The cystine cross-links are between such pairs.

Relation Between Urea-Bisulphite Solubility and Disulphide Exchange in Wool

H. Kessler and H. Zahn

Text. Research J., 28, 357-358 (April 1958) Experiments are recorded showing that mild alkaline pretreatment of wool fibres results in decreased ureabisulphite solubility for a virtually unchanged cystine-cysteine content. This decrease in solubility is held to support the assumption that intrachain disulphide groups are changed to interchain cross-linkages by the alkali. S.B.D.

Bilateral Structure and Swelling of Mildly Pre-treated Wools (Disulphide Exchange) G. Satlow and H. Kessler

Text. Research J., 28, 359 (April 1958) Microscopic examination with polarised light of untreated wool fibres when embedded in urea-bisulphite reagent shows that the bilateral structure appears after a few minutes owing to the decreasing birefringence of the orthocortex. If the fibre is given a mild alkaline pre-treatment the appearance of the bilateral structure is delayed, although the cystine content remains unchanged. Alkaline pretreatment also results in less swelling of the fibre when placed in the urea-bisulphite reagent. These observations are held to confirm the assumption that intrachain disulphide groups are changed to interchain cross-linkages by the alkali.

S.B.D.

Amino-acid Composition of Keratins. V—Comparison of the Chemical Composition of Merino Wools of Differing Crimp with that of Other Animal Fibres

D. H. Simmonds

Text, Research J., 28, 314-317 (April 1958)
The amino-acid compositions of two Merino wool samples having the same fibre diameter (22μ) but differing widely in the number of crimps per inch (9 and 15) are compared with those of a sample of human hair and one of kid mohair to determine whether their composition could be related to their chemical and physical properties. Out of 17 acids, differences were detected among the four samples in the cases of five-glycine, valine, leucine, cystine, and proline, the Merino wools, however, differed in only one case-proline. The results of the analyses do not conflict with the arrangement of the fibres according to the nature of their cortex and their chemical and physical properties—human hair, high-crimp wool, lowcrimp wool, and kid mohair.

Amino Acid Composition of Fractionated Cortical Cells from Wool

D. H. Simmonds and J. J. Bartulovich

Text. Research J., 28, 378-381 (May 1958) Prolonged cold acid treatment of wool enabled two cortical cell fractions to be separated by centrifuging.

Analyses carried out on the fractions showed that the heavier one was richer in 3 amino acids and deficient in 7 amino acids compared with the lighter fraction. Both fractions were richer than the original wool in 3 acids and deficient in 4 acids, the latter being concentrated in the material dissolved by the acid treatment.

Addition of Titanium Dioxide to the Polymerisation Mixture in the Manufacture of Polycaprolactam

E. Fritzsche and G. Odor

Faserforsch. und Textiltech., 9, 189-193 (May 1958) Titanium dioxide added during the polymerisation of e-aminocaproic acid acts as an accelerator and dispersing W.R.M. agent as well as producing a matt finish.

Structure and Phase State of Polyethylene Terephthalate Fibres

O. Gorbacheva and N. V. Mikhailov Colloid J. U.S.S.R., 20, 38–42 (Jan.-Feb. 1958) A study of polyester fibres by X-ray and thermographic analysis has shown that, in contrast to polyamide polymers, they easily supercool from the melt under the ordinary conditions of formation and therefore possess an amorphous structure. In the amorphous state polyethylene terephthalate is thermodynamically unstable, crystallisation of the polymer occurring relatively slowly at comparatively low temp. (80-110°c.). The heat of fusion is 9-11 cal./g.

PATENT

Man-made Fibres of Reduced Tendency to acquire an Electrostatic Charge Vereinigte Glanzstoff-Fabriken

Addition of 0·1-5·0% by weight of a substituted methane or ethane which forms a free radical containing trivalent hexaphenylethane and triphenylehloromethane, added to the monomer mixture used for preparing polycondensates or polymers results in filaments formed from such polycondensates or polymers having much less tendency to acquire an electrostatic charge than if the substituted methane or ethane had not been present.

Factors influencing the Crystal Structure of Cellulose Triacetate (XI p. 675)

Rabbit Fibres. XVIII - Frictional Properties of Angora Rabbit Fibres in Water (XII p. 676)

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Critical Detergent Concentration for Maximum Washing Efficiency

Textil Praxis, 13, 288-292 (March 1958)
Artificially soiled cotton and wool fabrics were washed under alkaline, acid, and neutral conditions using increasing amounts of non-ionic and anion-active detergents to determine critical concentrations for maximum washing efficiency. L.A.T.

Effects of Phosphates and Silicates on Raw Silk A. Hirata and M. Kobayashi

II- Effects of Condensed Phosphates on Raw Silk when treated with Water containing Ferrate J. Soc. Textile Cellulose Ind. Japan,

13, 797-800 (Dec. 1957)

The complex-forming ability with iron(III) of pyro- and tri-phosphate is greater than that of metaphosphate glass. The dissolving power of each condensed phosphate for sericin is proportional to the weight content. The dissolving power of the condensed phosphates decreases as the iron(III) contents increase. The effect of iron(III) removed from silk is obscure in the range of phosphate content < 0.01 g./200 c.c., but it is apparent at 0.5 g. per 200 c.c. in the order of pyro- and tri-phosphate; and the effect of metaphosphate glass has little relation to the weights added. In handle and appearance of the watertreated raw silk, triphosphate is better than the others, and sodium metaphosphate glass showed little or no effect; a mixture of soap and condensed phosphates is better than single additions. The depth of colour of the treated silk caused by iron(III) is proportional to the content of iron

(III), but it is less with a mixture of soap and condensed

III- Effects of Condensed Phosphates on Raw Silk when treated with Water containing Calcium Ion Ibid., 14, 37-39 (Jan. 1958)

The order of the softening ability of condensed phosphates for hard water is sodium metaphosphate glass (sodium hexametaphosphate) > sodium triphosphate > sodium pyrophosphate. The solvent power of water, soap, and silicate for sericin is decreased as the hardness of the water is increased respectively. The complex-forming ability of condensed phosphates for all metallic ions enables them to remove the contaminations of metallic scaps easily; therefore a mixture of soap and condensed phosphates is better than other degumming agents for raw silk when the hardness of the water is high. C.J.W.H.

Bleaching with Hydrogen Peroxide and with Peracetic Acid L. Chesner and G. C. Woodford

The methods and the theory of the bleaching of some of the principal textile fibres with hydrogen peroxide and peracetic acid are discussed. Observations are included on the greater ease of soil removal from bleached wool in relation to washing processes and the rôle of stabilisation in cotton-bleaching processes. Improvements to existing bleaching methods are given, with particular reference to the use of per acids in kier bleaching, and some data are presented on the decomposition of dilute solutions of peracetic acid. AUTHORS

New Developments in Sodium Chlorite Bleaching

Textil Praxis, 13, 298-304 (March 1958)

Inhibiting Corrosion by Chlorites

RP 795.690 Hercules Powder Co. Cellulose or a derivative thereof is treated with a solution of an inorganic chlorite or chlorine dioxide in a hot neutral slurrying medium containing a nitrogen compound, containing at least one H atom attached to the N atom, that will, at least slightly, dissociate in solution to yield ammonia or a basic volatile derivative of ammonia, e.g. NH_4Cl or $(NH_4)_2SO_4$. C.O.C. NH4Cl or (NH4)2SO4.

Washing Raw Wool and Similar Fibrous Materials (I p. 658)

VIII- DYEING

Dyeing Properties of Azo Acid Dyes—I M. Sekido and T. Iijima

J. Soc. Textile Cellulose Ind. Japan 14, 46-48 (Jan. 1958)

The dyeing properties of nine azo acid dyes having the same skeletal structure as α -naphthylamine $\rightarrow \beta$ -naphthol but different numbers and positions of the sulpho groups were determined. The absorption spectra of these dyes in the visible region are almost the same, except for dyes containing R acid as coupling component. The exhaustions (S.D.C. Method IV) of these dyes are proportional to the number of sulpho groups. Sulpho groups have no appreciable effect on the light fastness of these dyes.

Effect of Non-ionic Detergent on Direct Dyeings K. Nishida, K. Minekawa, and H. Uedaira

J. Soc. Textile Cellulose Ind. Japan

13, 893–898 (Dec. 1957) Time-absorption relations in the dyeing of silk and viscose rayon with the direct dye Diacotton Blue BB (C.I. Direct Blue 6) were determined. Also, the diffusion coefficients were obtained under the various dyeing conditions, by application of Crank's equation for a finite conditions, by application of Crank's equation for a finite dyebath. In the acid dyeing of silk, the amount of dye absorbed was very slightly decreased by the non-ionic detergent (Peletex LT 300, RO-[CH₂-CH₂-O]_R-CH₂-CH₃-CH₃-CH₅-CHO 5 g./litre). This effect was large in the neutral dyeing of silk and viscose rayon (bright). The experimental data on the acid dyeing of silk were in good agreement with the value calculated from Crank's equation.

These observations may be explained by taking into These observations may be explained by taking into account the interactions between dye, fibre, and dete C.J.W.H.

Changes in Vat Dye Systems G. S. Egerton and F. Galil

Nature, 181, 1599-1400 (17 May 1958) Colour changes sometimes observed when freshly vatdyed textiles are treated with boiling water or soap are caused by crystallisation of the dye. The absorption spectra of Cellophane sheets dyed with 1:5-dibenzamidoanthraquinone showed bathochromic shifts to wavelengths characteristic of the dye in the solid state. 1'-Acanaphthene-2-thianaphthalene-indigo behaved similarly, but here the spectral changes are also related to the cis-trans equilibrium. Both high light fastness and high washing fastness may arise from the presence of the vat dye in the fibre as the crystalline solid.

Dyeing of Cellulose Acetate. III—Comparison between Dyeing Mechanisms of Cellulose Acetate with Disperse Dyes and of Cellulose with Direct Dyes by the use of Heat of Dyeing and the change of Entropy of Dyeing K. Kusunose and K. Odajima

J. Soc. Textile Cellulose Ind. Japan, 14, 49-53 (Jan. 1958)

The heat of dyeing ΔH and the entropy change ΔS have been obtained for several commercial disperse dyes by the desorption method, and the data are compared with the corresponding data for the dyeing of collulosic fibres with direct dyes. There is a certain correlation between ΔH and ΔS when these quantities are plotted as abscissae ΔH and ΔS when these quantities are plotted as assumand and infinates, and the same correlation is found with direct dyes— $\Delta S = a + b\Delta H$ where a and b are constants. From these results it appears that the mechanism of the dyeing of cellulose acetate with disperse dyes is the same as that of the dyeing of cellulose with direct dyes. There is further discussion of the possible factors in dyes. There is further discussion of valoring dyeing processes other than hydrogen bonding.

C.J.W.H.

Reducing the Volatility of Aniline in Aniline Black Dyeing K. Ascik

Ochrona Pracy, (3), 91-95 (1954): Polish Tech. Abstr. No. 2, Abstr. No. 3544 (1954)

To obtain minimum volatility of the aniline it is best to use sparingly volatile aniline salts of strong acids, e.g. aniline hydrochloride, and the fabric should not be dried at > 50°c. or steamed at > 28°c.

Principles of Dyeing with Reactive Dyes T. Vickerstaff

Textil-Rund., 13, 267-282 (May 1958) A comprehensive review with formulae. S.R.C.

Behaviour of Reactive Dyes on Wool

F. Menchester

J.S.D.C., 74, 421-422 (May 1958)

Application of Azo Pigments to Nylon

A. Butterworth

milling, and rubbing.

J.S.D.C., 74, 480-481 (June 1958)

Chemical Modification of Fibres and Dyes H. Rath

Textil-Rund., 13, 282-292 (May 1958) The reactions of onium compounds are discussed.

8.R.C. Dyeing of Polyester Fibres with Vat Dyes in the Acid

Leuco Form H. Rummler

Textil Praxis, 13, 281-286 (March 1958) A number of vat dyes in their acid leuco form were found to be substantive to polyester fibres when applied at or near the boil at pH 6, particularly in the presence of swelling agents. After oxidation with hydrogen peroxide and subsequent soaping, the dyeings were treated in warm alkaline hydrosulphite to improve fastness to washing,

Dyeing Acrylic Fibres by the Cuprous Ion Method H. Rath and E. Specht

Melliand Textilber., 39, 281-282 (March 1958) It is suggested that cuprous complexes of aliphatic and aromatic nitriles can be used successfully in place of cupric salts and reducing agents during dyeing of acrylics with acid dyes. Methods of preparing such complexes and their reaction mechanism are described.

Reserve Effects on Arnel Cellulose Triacetate-Cotton Unions and Combinations with other Fibres W. A. Schoeneberg

Melliand Textilber., 39, 175-178 (Feb. 1958) Dye selection and dyeing and finishing procedures are described in detail. Vat and reactive dyes are recommended for the cotton component.

Dyeing of Fibres Difficult to Dye. IV-Condensationdyeing with an Active Halogen Compound and Free Amines. I

K. Yamada

Sen-i Gakkaishi, 12, 834-839 (1956): Chem. Abs., 52, 7706 (10 May 1958)

Condensation on the fibre of picryl chloride with free amines, particularly of the benzidine series and basic dyes containing a free NH₂ group, yielded even and fast dyeings-mostly reds and oranges. Thus fibre (1 g.) was stirred for 30 min. at 90°c. (60°c. for cellulose acetate) with 0.001n-amine hydrochloride (40 c.c.) and then with 0.001n-picryl chloride. This gave dyeings of the same colour value as those obtained with 10 times the amount of the intermediates condensed together before being applied to the fibre. The ease of dyeing of the various fibres is vinyl chloride > cellulose acetate > Vinylon and cotton > silk. Dyeing seems to be formation of a solid solution.

C.O.C. Light Fastness of Dyes and Dyed Textile Fabrics I. Rusznák and M. M. Fehérvári

Textil Praxis., 13, 293-298 (March 1958) Chemical changes in the dye and the substrate as a result of exposure to sunlight and artificial fading lamps have been investigated. Direct azo dyes were found to decompose into substantive and non-substantive components. Phenol and p- and o-quinones were the oxidation products of simple substances such as p- and o-aminoazobenzene respectively. After exposure to light, the dye uptake of cotton and viscose rayon was decreased and that of wool was enhanced. Prolonged exposure of cotton resulted in diminution of the D.P. and formation of numerous aldehydic groups. L.A.T.

PATENTS

Dyeing Wool with Sulphonated Copper Phthalocyanine BP 795,946 Ciba

Well levelled dyeings of good wet fastness properties are produced on wool with sulphonated Cu phthalocyanine from aqueous baths in presence of a compound containing at least one basic N atom to which is attached at least one residue containing a polyglycol ether chain and which also contains $\not<$ 4 C–C groups, e.g. the product obtained by condensing $\not<$ 4 mol. of an $a\beta$ -alkylene oxide with 1 mol. of an amine. C.O.C.

Dyeing Human Hair

Societe Monsavon-l'Oreal

The hair is treated with an aqueous alkaline composition containing 5:6-dihydroxyindole and ammonia, an ammonium salt or an amine, until the desired colour is obtained and then the hair is washed free of the composition. Usually 5:6-diacetoxyindole is used which hydrolyses to the 5:6-dihydroxyindole in the alkaline solution.

Dyeing Nitrogenous Fibrous Materials with Anthraquinonoid Acid Dyes BP 796,330 Ciba

Acid anthraquinone dyes containing an aromatically bound acylamino group (acyl derived from an aliphatic halogenocarboxylic acid), e.g. the dye-

readily dye wool, silk, leather, and polyamide or polyurethane fibres from an aqueous bath at pH 5.5-7.5.

Vat Dyeing of Cellulose Ethers and Esters

USP 2,798,788 Celanese Corpn. of America Cellulose acetate or the like dyed with a dispersion of a vat acid in presence of polyvinyl pyrrolidone is level, well penetrated and of good fastness to rubbing.

Dyeing of Polyester Fibres

BP 795.768 Societe Rhodiaceta The fibres are treated with an azoic or diazo component dispersed in a solution of an alkylanilide or alkoxyanilide of β-hydroxynaphthoic acid at pH 8.5-10.5 and 100°c. They are then heated in a diazotising bath at 20-85°c. This enables use of baths as short as 5:1 on the weight of goods. It is advisable to treat the dyed material in a reducing bath (hydrosulphite) before drying. C.O.C.

Vat Dyeing of Polyester Fibres

BP 795,392

ICI Greatly increased affinity is obtained when polyester fibre is treated under pressure at 100-200°c. with an aqueous suspension of a reduced vat dye at pH 5-9.

Colouring Dry or Powdered Foods with Caramel (C.I. Natural Brown 10) or other Food Dye

Addition to a dry powdered food of 1-7% of dry C.I. Natural Brown 10 or other food dye together with 0.25-1.1% of a water soluble cellulose ether yields a product which is evenly and deeply coloured when it is mixed with C.O.C.

Dyeing of Wool with Lichens and Lichen Substances (IV p. 662)

Transfer of Disperse Dyes to Secondary Cellulose Acetate during Steaming (IX below)
Photo-oxidation of Indigosols on Rayon (IX below)

Finishing of Nylon Textiles (X p. 672)

Improving the Penetration of Cellulosic Fibres by Dyes or Finishes (X p. 672)

Imparting Improved Dyeing Properties and an Anti-static Finish to Hydrophobic Textiles (X p. 672)

Wet Processing of Textiles on Perforated Beams (X p. 672) Pile Fabric (X p. 673) Coated Fabrics—Manufacturing Processes and Pigments

Used (XIII p. 676)

IX-PRINTING

Transfer of Disperse Dyes to Secondary Cellulose Acetate during Steaming E. H. Daruwalla and V. R. Limaye

J.S.D.C., 74, 464-475 (June 1958) The work reported by Daruwalla and Turner (ibid., 69, 240 (1953)) has been extended to include a study of the transfer of selected aminoanthraquinone and aminoazo disperse dyes to secondary cellulose acetate. The effects of particle size and chemical structure of dye, presence of dispersing agents, and different thickener compositions have been examined for appropriate conditions of application. With the majority of the dyes studied a constant partition of dye between cellulose acetate phase and thickener phase at equilibrium is maintained with increasing proportions of dye in the system until saturation of the cellulose acetate with the dye is reached. Study of binary mixtures of selected dyes indicates that the transfer characteristics of one component are unaffected by the presence of the other. The results are discussed with special reference to quantitative studies by other workers on the behaviour of these dyes during dyeing.

Photo-oxidation of Indigosols on Rayon

A. Polster

Magyar Kem. Folyóirat, 61, 296-297 (1955):

AUTHORS

Chem. Abs., 52, 9603 (10 June 1958) Indigosol Red Violet IRH (C.I. Solubilised Vat Violet 2) and Indigosol O4B (C.I. Solubilised Vat Blue 5) transform into Indigo upon oxidation under illumination with light of 280-350 mµ. Thus to solubilised vat dye (100 g.) in water (10 l.), brought to pH 3.2-3.4 with formic acid, were added Na₂SO₄ (66 g.), K metabisulphite (6 g.), NH₄ vanadate $(0.07~{\rm mg.})$, thiourea $(15~{\rm g.})$, urea $(15~{\rm g.})$, glacial acetic acid $(5~{\rm ml.})$, conc. formic acid $(5~{\rm ml.})$, and Na acetate $(75~{\rm g.})$. The rayon was steeped in the liquor, squeezed, covered with a negative and exposed for 5-10 min. to a Hanan S-300 mercury vapour lamp. Indigo was formed in proportion to the absorbed light energy. The exposed rayon was then soaped, boiled in aqueous alkali and ironed. Increase in the amount of vanadate accelerates the reaction but this is undesirable because the system will tend to overoxidise and cause the colouring not to be dependent on the blackness of the negative. The colour thus obtained is fast to light, rubbing and hot ironing.

Discharge Printing of Polyester Fibres

G. Bernardy and H. Wirth Melliand Textilber., 39, 292-297 (March 1958) Existing methods of printing polyester fibres are reviewed, and a new process for the production of coloured discharge and discharge resist effects is described. The novelty lies in-(1) application of insoluble zinc formaldehyde-sulphoxylate as a discharging agent, (2) use of stannous chloride and anthraquinone to strengthen the discharging effect of soluble zine formaldehyde-sulphoxylate, and (3) simplification of the discharge reserve process by reversing the usual order of procedure and applying the cover printing dye in the form of an oil-in-water emulsion. L.A.T.

PATENTS

Plural Colour Doctor Knife for Use with Textile **Printing Machines**

W. P. Ros USP 2,800,075 A doctor knife wherein the different printing pastes run through passages closely parallel to one another with positive assurance that they will not contaminate one another as they are printed onto the fabric. C.O.C.

Printing Pastes, Sizes, Films, etc. USP 2,800,417

Insoluble coatings useful, e.g. for fixing pigments in textile printing, for producing films or imprognating or textile printing, for producing initis or interegnating or sizing, are obtained by using soluble or fusible substances containing both basic N groups capable of salt formation and groups which can react with the basic N groups to effect cross-linking. Thus a polymer of butyl acrylate of K-value 20 is refluxed with tetramethylene diamine in butanol until the product freed from solvent is soluble in dil. acetic acid. The solvent and excess amine are distilled off in vacuo and the residue well washed with water several times, neutralised with acetic acid and made into a 40% aqueous paste. This is then mixed with gum tragacanth, water and Cu phthalocyanine (C.I. Pigment Blue 15), printed onto cotton and heated at 140°c. for 30 min. This yields full blue prints fast to washing, boiling soda and

Copy Production particularly by Hectographing D. J. O'Sullivan and E. Lefebure BP 793

Impressions are made on paper by a composition containing a colourless complex of a dye, reduced dye or dye intermediate with a non-ionic salt-free surface-active agent. A second sheet of paper is coated with or contains an oxidising agent which can react with the complex in the first paper to form a colorant. The two sheets are then pressed together to produce coloration on the sheet containing the oxidising agent at those points corresponding with the impressions on the complex-coated sheet. The principle can be applied to hectographing by incorporating the complex with waxes and/or waxy materials in the coating used and the oxidising agent is incorporated in the liquid used in the reproduction process for damping the paper on which the copy is to be produced.

C.O.C.

Colour Couplers BP 797,141 Developing a reducible silver salt image with a primary aromatic amino developer in presence of a 2:5-diacyl-

aminophenol (acyl derived from a carboxylic acid), e.g. 2-acetamido-5-[2:4-di-tert.-amylphenoxyacetamido]phenol, yields eyan dyes of low blue light absorption and a high degree of heat stability. C.O.C.

Coloured Photographs by Colour Development Agfa AG für Photofabrikation BP 795,476

Use as colour developer of 1-(p-aminophenyl)-3-aminopyrazoline or a derivative obtained by introducing a substituent into the 1-phenyl nucleus yields very rapid and intensive coupling. The colours obtained are very different from those obtained with the usual p-phenylenediamine developers.

Colour Photography by Colour Development

Agfa Addition to the colour developer of 1-(p-aminophenyl)-3aminopyrazoline or a derivative in which the H atoms of the p-amino group in the 1-phenyl radical and/or of the 1-phenyl nucleus itself are replaced by at least one substituent enables less colour developer to be used or the time of development shortened while obtaining the same colour intensities and colour gradations.

"Dupe" Negatives in Colour Cinematography
BP 795,186

BP 796,054 A method, using two or three layer colour negative

materials, of preparing commercially satisfactory duplicate (so-called "dupe") negatives. C.O.C.

Pyridine-2:6-dicarboxylic Acids as Sequestering Agents in Photographic Elements or Processing Baths (III

X—SIZING AND FINISHING

Resin Distribution in Resin-finished Fabric. III—Migration of the Bath Solids during the Pre-drying 8. Yokoyama

J. Soc. Textile Cellulose Ind. Japan, 14, 96-99 (Feb. 1958)

In the finishing of cellulosic fabrics with precondensates of thermosetting resins, migration of bath solids occurs only during the initial stages of pre-drying. It has a marked effect on the distribution of resin.

Crease-resist Finishing T. Poros

Magyar Textiltechnika, (1), 44-47 (1957): Hungarian Tech. Abs., 10, (1), 18 (1958)

The idea underlying the improvement of the technology of crease-resistant cotton finishing is to increase the amorphous portion of the fibre, i.e. the part accessible to the preliminary-stage condensate. The structure of the natural fibres is made to resemble that of man-made fibres, whereby damage due to mechanical action may be reduced. Tests were conducted on cotton fabrics previously swelled with soda lye and treated with urea-formaldehyde resins under works conditions. Maximum increase of permanent elongation and maximum decrease of elastic elongation were attained at a lye conen. of 180-200 g./litre. Increasing lye conen. entailed increasing resistance to wear, which in turn was considerably reduced by increases in the concentration of the resin. A dimethylolurea preliminary-stage condensate was used for the creasuresisting finishing of diverse cotton fabrics. Tensile strength was impaired by increasing the resin concentration. However, this reduction in tensile strength could be counteracted by adequate preliminary lye treatment. The decrease in tensile strength was less when the cotton was treated in the lye in a loose state than after mercerisation. A drawback of the preliminary lye treatment, however, is that it reduces the crease angle values characterising C.J.W.H. elasticity.

Bisulphite as an Agent for Binding Free Form-aldehyde in Crease-resist Finishing Baths J. Majzner, Z. Jędrusiak, and J. Brzeziński

Przemysł Włókienniczy, (6), 285–289 (1957):
Polish Tech. Abs., 29, (1), 143 (1958)
The urea-formaldehyde condensate used in the textile

industry under the name of "Antimnol FM" contains immediately after synthesis 2.5%, and after 5 months' storage still 1.4%, of free formaldehyde. Bisulphite has certain advantages over numerous other capable of serving as acceptors for free formaldehyde, the maximum concentration of which in the air ought not to exceed 0.005 mg./litre. Laboratory investigations checked on an industrial scale, demonstrated that an addition of technical bisulphite (30-45 g./litre) to an Antimnol FM bath (350 g./litre) produced optimum conditions of work without noticeably impairing the properties of the material treated. However, bisulphite removes to any large degree only formaldehyde vapours, whereas pyridine vapours released by Petefobol IW, a softening and hydrophobic agent, still remain a threat to the health of workers. Control analyses of air samples from different places, taken twice at 15-min. intervals after 4-5 hr. of continuous proofing, demonstrated that the concentration of free formaldehyde in the air over the trough of the padding machine was, without bisulphite addition, higher than is admissible, and dropped, after addition of bisulphite to the proofing bath, to one-tenth of the permissible level. It was also found that, when bisulphite was added to the impregnating bath, the formaldehyde content in the air was lower than the admissible maximum throughout C.J.W.H.

Infrared Study of Resin-treated Cellulose Hydrate Films

W. Bandel

Melliand Textilber., 39, 204-211 (Feb. 1958) From the spectrographic evidence in the infrared region it is confirmed that urea, melamine, polyvinyl, and epoxy resins form chemical links with cellulose, and this factor determines the washing fastness of the various resin L.A.T.

Resin Finishing with Acrylic Acid Monomers. II— Influences of Concentration of Hydrogen Peroxide, Time, and Temperature on Yield of Polymer 8. Yamada and H. Kojima

J. Soc. Textile Cellulose Ind. Japan,

14, 106-110 (Feb. 1958) Certain physical properties of spun rayon fabric are improved by intra-fibre polymerisation of monomeric CH₂:CH·COOH. With 0.76-2.68 m. monomer the conen. of H₂O₂ should be 0.0736 m. and the mixture should be heated at 110°c. for 1 hr. Polymer yields vary considerably between 80° and 110°c., but only small differences are noted between 100° and 120°c. The duration of poly-merisation is practically independent of monomer concentration. The more dilute the solution the larger is the loss due to evaporation and the more rapid is decom-

C.J.W.H. Polymerisation of Acrylic Esters in Cellulosic Fibres. I—Polymerisation of Methyl Acrylate in Allylated Cellulosic Fibres H. Tonami

position of H₂O₂, which is quite small up to 30 min.

J. Soc. Textile Cellulose Ind. Japan. 14, 100-105 (Feb. 1958)

A new process for imparting wrinkle resistance to cotton and viscose rayon fabrics entails formation of cross-links by methyl acrylate resin within fibres. There are two stages-firstly allylation of cellulosic fibres by NaOHallyl bromide treatment, and secondly polymerisation of methyl acrylate monomer in the allylated fibres. The formation of cross-linkages was shown by insolubility in Schweizer's reagent and the stress strain properties in dry and wet states. The treated fabrics had good wrinkle recovery, but their breaking strength was considerably C.J.W.H. decreased.

Organo-tin Compounds as Textile Preservatives H. J. Lueck and J. G. A. Luijten

J.S.D.C., 74, 476-480 (June 1958) The suitability of a number of organo-tin compounds as preservatives for textiles has been investigated. The investigations comprise tests on the mothproofing and earpet-beetleproofing of wool and on the rotproofing of jute and cotton. The tests have been combined with experiments on fastness to washing and with leaching experiments. AUTHORS

Reaction of Formaldehyde with Wool and its Effect on Digestion by Insects J. R. McPhee

Text. Research J., 28, 303-314 (April 1958)

The effect of treatment with formaldehyde soln. at varying pH on the amino-acid residues in wool protein is examined. Under optimum conditions the wool can be made resistant to attack from larvae of the clothes moth (Tineola) and from newly hatched but not fully active larvae of the carpet beetle (Anthrenus). Possible mechanisms for this effect are discussed. The treated wool shows increased resistance to alkali, acid, thioglycollate, and trypsin solutions, and to soil micro-organisms. The appearance, felting shrinkage, bursting strength, abrasion resistance, and yarn strength are unaltered.

Finishing of Nylon Textiles VI—Effects of Tension and Cooling on Heat-setting T. Yasuda

J. Soc. Textile Cellulose Ind. Japan.

14, 40-43 (Jan. 1958) The internal structure of nylon fibres is affected by tension changes, especially in the degree of molecular arrangement, even at the same temperature, and these changes influence their physical properties. Cooling condition, too, has evident bearing upon their physical properties. But a crystal structure was not seen by rapid cooling. These results indicate that it is very important to control tensile and cooling conditions carefully during the setting of nylon.

VII- Effects of Tensile Hysteresis on the Dyeing

Properties T. Yasuda and Y. Satō

Ibid., 43-45

The rate of dyeing of fibres which have been stretched and then relaxed becomes very low, though the saturation amount of dye at equilibrium does not differ greatly. Tension applied in weaving has a very important bearing upon dyeing properties, and tensile hysteresis of nylon fibres seems to be one of the causes of "stripe". C.J.W.H.

Acetalisation of Polyvinyl Alcohol N. Nakamura

VIII-Formalisation after Nonanalisation in Two Stages and Simultaneous Mixed Acetalisation in a Single Stage

J. Soc. Textile Cellulose Ind. Japan, 13, 823-825 (Dec. 1957)

Formalisation of polyvinyl alcohol fibre after nonanalisation takes place smoothly, and fibres with various degrees of mixed acetalisation may be easily obtained. Simultaneous mixed acetalisation is difficult, for methanol must be used as a solvent for nonanaldehyde, and in methanol solution the nonanalisation predominates.

IX- Relation Between Resistance to Boiling Water and Degree of Nonanalisation

About 200 samples of nonanalised fibres and of fibres acetalised with a mixture of nonanaldehyde and formaldehyde were tested for shrinkage in boiling water. the degree of acetalisation is as high as 15-25 mol.% the resistance to boiling water was adequate with both single and mixed acetalisation, but in the latter case the degree of nonanalisation must not be < 5%.

PATENTS

Improving the Penetration of Cellulosic Fibres by Dyes or Finishes

National Lead Co. Better penetration of cellulosic fibres by dyes, sizes, finishing agents, etc. is obtained if the fibres are treated either previously or simultaneously with a titanium halide of valency < 4, e.g. TiCl₃.

Permanent Sizing of Coloured Cellulose Acetate Warps USP 2,799,914

An aqueous solution of an ammonium salt of a copolymer of 95-99% vinyl acetate and 5-1% of an ethylenically unsaturated aliphatic compound of 3-8 C and containing a carboxy group, e.g. crotonic acid, is used. The size on the yarns is unaffected by mild laundering or dry cleaning. C.O.C.

Imparting Improved Dyeing Properties and an Anti-static Finish to Hydrophobic Textiles BP 797,175 Onvx Oil & Chemical Co.

The water-soluble addition products of aliphatic polyamines containing > 1 primary amine group with poly-alkoxy compounds applied to hydrophobic textiles impart an antistatic finish which is fast to washing and dry cleaning. Thus undyed unbleached polyester taffeta is impregnated with an aqueous solution of the diiodide of polyethylene glycol (mol. wt. 820) and diethylene triamine, dried at 50°c, and baked at 150°c. This gives an antistatio finish which is resistant to repeated laundering. The treated fabric is readily dyed with acid or other dyes having affinity for wool. C.O.C.

Wet Processing of Textiles on Perforated Beams E. E. Bellmann, R. Kronsbein, and F. Bellmann

On either side of each end of the piece there are affixed strips of impervious material extending outwards from the selvedge. When the piece is batched each of these strips forms a closed roll and their free ends are fastened to the beam. This makes the strips fastened to the same edge of

the piece form a hollow sealing space which yields to shrinking of the batched material. C.O.C.

Finishing Cellulose Triacetate Cloth

BP 795,821 The handle of cellulose triacetate cloth is greatly improved by treating it with a liquid softening agent, e.g. 52% acetic acid, until it has shrunk ≮ 5% warpways measured after removal of the softening agent and drying.

BrC BP 797,135 The pile contains short lengths of twisted yarn made partly or wholly of man-made fibres and whose twist has been set by increasing the degree of crystallinity of the man-made fibres. This increase of crystallinity may be brought about, e.g. by heating the pile yarn either before or after weaving or by treating the yarn with a suitable solvent, e.g. aqueous acetone. In the latter case incorporation of a dye into the solvent enables dyeing to be carried out at the same time C.O.C.

Fabric having Undulation Effects

BP 796,933 Heberlein & Co. Fabric having undulation effects, e.g. smocked crêpe fabrics, are obtained by using in the weft and/or warp highly elastic yarns made of crimped man-made filaments which are potentially shrinkable while preserving their elasticity. The fabric is wet shrunk in absence of tension in the highly elastic yarns. C.O.C.

Water-repellent Finish Monsanto Chemicals

BP 796,140-1 Porous materials, e.g. textiles, are rendered water-repellent by treating them with an aqueous solution of a lead or thorium salt or an alkali metal siliconate, and with a silicone dissolved in an organic solvent containing a polymerisation catalyst. The solutions are applied in either order. C.O.C.

Metallising Textiles Heberlein & Co.

Textiles, especially cellulosic textiles, which have been coated by depositing metal from the vapour state on them in a high vacuum have the adhesion of the metal to them considerably increased if the coated material is heated at C.O.C.

Decorative Effects by Metallising Pliable Sheet Materials

Heberlein & Co. BP 796,139 Pliable sheet material having pronounced projections and cavities, e.g. textiles, is coated with metal by causing metal vapour to impinge upon it at an acute angle to its surface. This causes the coating to be non-uniform, being thicker on one side of the projections than on the other side. C.O.C.

Vitaminised Cloth

BP 795,557 R. H. Monceaux The material is impregnated with a solution or finish containing vitamin P. The treated materials have a vaso-constricting effect when worn next to the skin. C.O.C.

Finishing of Asbestos Cloth

United States Rubber Co. The cloth is thoroughly wet out, brushed on both faces, and then dried preferably without squeezing any water out of it. The dried cloth has greatly enhanced lustre and whiteness, appears to be of a much finer weave and is more C.O.C.

Addition of Titanium Dioxide to the Polymerisation
Mixture in the Manufacture of Polycaprolactam

Fibres (VI p. 668) Effect of Water Removal on the Crystallinity of Cellulose

(XI p. 674) Coated Fabrics - Manufacturing Processes and Pigments used (XIII p. 676)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Relationship between Brightness Reversion and Carbonyl Content in Cellulose Pulp I. Jullander and K. Brune

Acta Chem. Scand., 11, 570-571 (1957):

Chem. Abs., 52, 8547 (25 May 1958)
The brightness reversion of cellulose (expressed as "post-colour" value calculated by Geitz's method (Chem. Abs.,

39, 3927 (1945)) was determined for several samples whose CO content (measured by Cu no.) varied progressively by reduction with increasing amounts of NaBH₄. Thus airdried pulp was agitated and mixed with 0·1-10·0% NaBH₄ (based on the pulp which was present in 2% concentration) for 3 hr. at $<50^{\circ}$ c. The pulp was then washed, acidifed with acetic acid, washed with water to pH 6.5 and airdried. Brightness measurements of sheets made from these samples were plotted against Cu number. CO groups were responsible for about half the brightness reversion for several types of pulps. Highly refined sulphite pulp retained its brightness as well as did cotton linters. C.O.C.

Wet Strength of Paper

I. Yoshino

XIV-Development of the Wet Strength of Papers coated with some High Polymers by Heat Treat-J. Soc. Textile Cellulose Ind. Japan,

14, 8-11 (Jan. 1958) By heat treatment papers coated with some watersoluble high polymers develop wet strength more markedly than in the uncoated paper. The most effective polymer is a protein like gelatin or milk casein, followed by carboxymethylcellulose or polyvinyl alcohol. The reason why protein is most effective seems to be the stronger protonaccepting power of the functional groups in a protein molecule, and the lower activation energy of adhesion. The latter may be attributed to the special character of the functional groups and molecular structure. Carboxy-methylcellulose contains carboxyl groups with strong proton-accepting power. Polyvinyl alcohol possesses in addition to its polar hydroxyl groups rather high ability of free molecular rotation. It appears therefore that high polymers which form hydrogen bonds easily with cellulose fibre are effective in increasing the wet strength, i.e. a class of polymers having low activation energy of adhesion. The high ability of molecular free rotation, absence of sterio hindrance, and strong proton-accepting power of functional groups are also important factors.

XV-Influence of the Various Polar Media on the Fibre Bonding of Paper

The zerospan tensile strength, tensile strength and loadelongation curve of paper are determined, after it has been soaked in six liquids—water, glycerol, monoacetin, diacetin, triacetin, decalin—at room temperature and at 180°c. and the liquids have been replaced by water. The results obtained at room temperature show that the dry strength of the untreated paper depends more on the bonding between fibres than on the original strength of the fibre, and that the higher the cohesion energy density of the liquids, the more readily they break the bonding. results obtained at 180°c. show that the fibre bonding, which causes the development of the wet strength of th heat-treated paper, is effectively formed in the media of lower cohesion energy density. These results seem to support the interpretation that this fibre bonding is due to formation of hydrogen bonds between fibres

XVI-Physical Properties of the Heat-treated Ibid., 92-95 (Feb. 1958)

When paper is subjected to heat treatment at 180-200°C. the wet strength is markedly increased, and other physical properties also change. The dry tensile and burst strength are increased, and the tear strength is reduced. elongation of the wet paper is greater with prolonged heat treatment, but the irreversible elongation is increased only during the initial period of heating, and soon becomes almost constant. In comparison with the untreated paper, the dynamic Young's modulus of the heat-treated paper is scarcely increased in the dry condition, but is increased in the wet condition. Determination of the impact strength and the moisture expansivity show that the heat-treated paper is very insensitive to the influence of water and moisture. The electrical puncture voltage of paper is increased and the power factor of it decreased with the C.J.W.H. time of heat treatment.

Some Variables in the De-inking of Waste Paper

P. Kajanne and V. Östring Paperi ja Puu, 40, 201-206

(Special issue 4s, April 1958)
The matured test sheets are immersed in thermostatic baths containing varying amounts of different chemicals

and stirred by a stroboscopically controlled device directing the medium towards the ink stamp. Powdered plastic is used as a mechanical rubbing agent to simulate the action of the fibres in the pulp. The effects of NaOH, Na silicate, and Na,O, on the resulting whiteness, and their discolouring action upon unbleached cellulose and pulped newspaper, vary considerably. Surface-active agents have both beneficial and adverse effects, while softening agents exert only adverse effects. There is a positive correlation between washing temp. and amount of ink removed, but the advantage is somewhat offset by the fact that pulps are more discoloured at high temp.

Systemic Absences in the X-Ray Diagram of Cellulose K. C. Ellis and J. O. Warwicker

Nature, 181, 1614-1615 (7 June 1958) Many X-ray diffraction photographs from high polymers show only even-order meridional reflexions, leading to the assumption of a twofold screw axis in the structure. It is pointed out that absences of reflexions may occur if the structure is regarded as built up of two interlinked lattices, the absent reflexions being associated with the identity of two lattice units along the y axis. In polymers where the asymmetric units consist of parts of more than one chain this condition can be attained by displacing half the chains by half a cell division relative to the rest and at the same time allowing them to rotate through arbitrary angles about the chain axis. There is thus no a priori reason to assume a twofold screw axis in the chain or structure of cellulose. Other configurations may be postulated which have hitherto been neglected. One of these is briefly discussed. The effect on the unit cell volume is also considered, and it is necessary at least to double it to comply with other structural features. W.R.M.

Effect of Water Removal on the Crystallinity of Cellulose

M. Kouris, H. Ruck, and S. G. Mason

Canadian J. Chem., 36, 931-948 (June 1958) The effects of various methods of drying on crystallinity has been studied by X-ray diffraction. No change was observed when wood cellulose fibres were dried from water under different conditions, but a lower crystallinity was obtained when they were dried from benzene. Heats of wetting suggest changes in accessibility with different drying methods. Crystallinity is substantially reduced or obliterated by milling. Wetting with water and drying restored the crystallinity. Other liquids gave less definite results, but it was concluded that alcohols reform the lattice to a limited extent. W.R.M.

Hydrolysis of Cellulose and its Relation to Structure $-\Pi$

A. Sharples

Trans. Faraday Soc., 54, 913-917 (June 1958) Results of studies on the hydrolytic degradation of cellulose crystallites are discussed in relation to possible mechanisms of hydrolysis. An inverse relation is found to hold between the hydrolysis rate constant and the mean crystallite length, and from this it is confirmed that the mechanism is one involving end attack. The results also indicate that the mean length of the crystallites is related to the amount of crystalline material present.

W.R.M. Effect of Velocity Gradient and Cellulose Concentration on Viscosities of Cellulose Solutions

M. Horio, S. Onogi, Y. Taniguchi, T. Kobayashi, and H. Kuroda

J. Soc. Textile Cellulose Ind. Japan,

13, 830-836 (Dec. 1957) The flow curves of three kinds of cellulose materials such as purified cotton and wood pulps in cupriethylene-diamine solution (CED) and cuprammonium solution (CAM) have been measured over a wide range of velocity gradients. The viscosity measurements were carried out by employing a capillary viscometer with continuously varying pressure head, which is a modification of the viscometer described by Maron, Krieger, and Sisko. The solvents themselves behave as Newtonian liquids, whereas the cellulose solutions manifest non-Newtonian behaviour unless the concentration is extremely low. For concentrations $< 3/[\eta]$ the wood pulps in CED behave practically as Newtonian liquids, and therefore it seems that the intrinsic viscosity may be satisfactorily determined from a single point. In the case of celluloses having higher molecular

weights, such as cotton, much more dilute solutions must be used in order to apply the single-point method. Although the concentrated solutions exhibit marked non-Newtonian behaviour, the apparent viscosities become constant at low velocity gradients. Hence measurement of intrinsic viscosity by the single-point method should be made on a dilute solution at as low a velocity gradient as possible. Even the most dilute solution of a dissolving pulp for viscose in CAM (0.522 g./100 ml.) in the present work exhibits non-Newtonian behaviour, suggesting that the current method (JIS P 8101) must be employed carefully for determining the relative viscosity of cellulose having a high D.P. C.J.W.H.

Alkaline Solutions of Iron-Tartaric Acid Complex as Solvents for Cellulose G. Jayme and W. Bergmann

Das Papier, 12, 187-196 (May 1958) A good solvent for cellulose is prepared from stoichio-metric proportions of ferric nitrate, tartaric acid, and caustic soda. The ppt. is filtered off, washed, dried, and added to the calculated amounts of tartaric acid and caustic soda, giving a clear green stable solution of the complex sodium "ferritartrate". Glycerol is a convenient agent for precipitating cellulose from the complex solution, thus allowing fractionation of pulps. The solvent is useful for D.P. measurements, and two methods are described, one applicable to pulps of very high D.P. values.

Adsorbed Metal Ions on the Surface of Cellulose Monofibrils

D. S. Belford, A. Myers, and R. D. Preston

Nature, 181, 1516-1518 (31 May 1958) Electron diffraction studies show that metal-cellulose complexes are formed when cellulose is treated with dilute aqueous salt solutions. The metal is adsorbed on the surface of microfibrils. Quantitative estimation of bound copper shows that adsorption conforms to a Langmuir adsorption isotherm. The metal atoms are oriented, implying a high degree of order of cellulose molecules at the surface of the microfibril. W.R.M.

Reaction of Alkali Celluloses with Carbon Diselenide E. Treiber and J. Rehnström

Das Papier, 12, 274-277 (June 1958) The rapid reaction leads to a very unstable product of extremely low solubility. Spectroscopic examination shows this product to be a selenium compound corresponding to cellulose xanthate. Even at -5°c. alkali cellulose quickly becomes dark yellow and turns into a reddishbrown fibrous product. At ordinary reaction temp. of approx. 25°c. this happens within a few minutes. R.A.

Hydrodynamic Characteristics and Degree of Poly-

dispersity of Ethylcellulose T. I. Samsonova and S. Ya. Frenkel'

Colloid J. U.S.S.R., 20, 67-78 (Jan.-Feb. 1958) The hydrodynamic properties and the degree of polydispersity of typical industrial specimens of ethylcellulose, as well as the effect of oxidation on these properties, have been studied. All specimens had a relatively low mol. wt. (< 70,000) and possessed a considerable degree of polydispersity. It is found that oxidation is accompanied by depolymerisation and a change in the character of polydispersity. The extent of substitution and the change in the chemical composition caused by the oxidation have no apparent effect on the hydrodynamic behaviour of the macromolecules of ethylcellulose. The effective length of an ethylcellulose chain unit in ethyl acetate is 25 A., being half that for nitrocellulose. In the course of the oxidation there is a sharp fall in the mechanical strength and in the relative elongation of films prepared from the corresponding samples.

Heterogeneity of Substitution in Hydroxyethylcelluloses

O. A. Battista, C. W. Tasker, and R. T. K. Cornwell

Svensk Papperstidning, 61, 272-275 (15 May 1958) The investigation was carried out on a yarn spun from a commercial sample of hydroxyethylcellulose having a degree of substitution of 0.14, 0.13, which was progressively etherified further by treatment in skein form (1.955 g.) with ethylene oxide (15g.) at 90°c. in a steel autoclave. Relative viscosity measurements, expressed in terms of apparent basic D.P., can be used to characterise the heterogeneity with which ethylene oxide polymerises or grafts on to the cellulose molecule. In the case of low-D.S. or alkali-soluble hydroxyethylcelluloses, measurement of % hydrolysis-resistant residues may also serve as a parameter for following this heterogeneity. The accessibility of the fine structure of water-insoluble hydroxyethylcelluloses to hydrolytic attack at a given mean substitution should be related to the length of the polyoxyethylene chain build-up and the distribution of such side-chains along the cellulose

Carboxymethylcellulose. II- Preparation of Sodium Carboxymethylcellulose by Multistage Etherification E. Hayakawa and Y. Morita

Rept. Govt. Chem. Ind. Research Inst. Tokyo,

53, 37-75 (vii-viii) (Feb. 1958) Double-stage etherification has no advantage over single-stage in the preparation of sodium carboxymethylcellulose of degree of substitution (D.S.) < 1.0 but has considerable advantages in the preparation of material of relatively high D.S. (1.5-2). Purification and the use of as small a quantity of chloroacetic acid as possible in each stage are important. It is concluded that there is no appreciable difference in reactivity among the three hydroxyl groups of the glucose unit and that the reaction is heterogeneous in various aspects, involving both intermicellar and intramicellar reactions. In the earlier stages the intermicellar reaction may predominate; in later stages intramicellar reactions may be of a quasi-homogeneous or permutoid type. W.R.M.

Effect of the Acetyl Group Content of Cellulose Acetate on the Properties of its Solutions V. P. Kharitonova and A. B. Pakshve

Colloid J. U.S.S.R., 20, 110-116 (Jan.-Feb. 1958) With a change in the acetyl group content of cellulose acetate the viscosity of its solutions in various solvents changes, passing through a min. when the content is 56.5-58.5%. The latter is explained by the max. flexibility of the macromolecules in the solution owing to the least regular arrangement of the polar hydroxyl and acetyl groups. The heats of solution depend upon the ratio of acetyl to hydroxyl groups and decrease on passing from formic acid to acetone. The turbidity of solutions of cellulose acetate of the same composition and mol. wt. changes 100-fold in different solvents. The addition of small amounts of a second component which causes solvation of the polar groups of cellulose acetate diminishes the turbidity by 40-60%. As the fractions of low acetyl content increase in industrial cellulose acetate, the G.J.K. turbidity of the solutions increases.

Factors influencing the Crystal Structure of Cellulose Triacetate

B. S. Sprague, J. L. Riley, and H. D. Noether

Text. Research J., 28, 275-287 (April 1958)
The effects of source of cellulose and conditions of acetylation on the crystalline form of cellulose triacetate are examined. The use of heat treatments enables the existence of two stable polymorphic crystalline forms, triacetate I and triacetate II, to be demonstrated. Conditions for transforming triacetate I into triacetate II are discussed, but the possibility of the reverse transformation is questioned. The action of swelling agents on the ability of each form of triacetate to attain high lateral order is investigated, and a possible mechanism proposed. Suggestions are made for the unit call dimensions of triacetate I tions are made for the unit cell dimensions of triacetate I and triacetate II, these being compared with cellulose I and cellulose II structures.

Molecular Size and Configuration of Cellulose Trinitrate in Solution M. M. Huque, D. A. I. Goring, and S. G. Mason

Canadian J. Chem., 36, 952-969 (June 1958) Viscosity and light-scattering measurements are made on solutions of fractionated and unfractionated samples of cellulose trinitrate in acetone and ethyl acetate. The range of molecular weight is 6.5-25 × 10s. The results suggest that random coil configuration is attained in acctone but that a configurational transition occurs in ethyl acetate. The values of the exponent a in the relationship between intrinsic viscosity and molecular weight are less than unity intrinsic viscosity and moreouser weight and approximately the same in the two solvents.

W.R.M.

PATENTS.

Highly Hydrated, Degasified Papermaking Fibres Zellstoffverfahrens

The aqueous suspension of fibres is passed through an ultrasonic field of 22-800 kilocycles per sec. at fibre consistencies between 0.8 and 0.1%, while keeping flocculation to a minimum, until maximum tearing strength is established. An exceptionally high increase in hydration value takes place without appreciable deterioration in drainage capacity or fibre length. At the same time degasification of the actual fibres takes place, both internally and on the surface. In conjunction with, and preferably after, the ultrasonic treatment, the fibres are subjected to gentle beating, using beater units without cutting edges and coated with a resilient material, e.g. rubber, so that beating is carried out with a squeezing effect rather than a cutting action. The heat evolved effect rather than a cutting action during ultrasonic absorption is removed by cooling.

High-gloss Printing Paper John C. Rice

BP 796,934

Paper is coated with a composition high in fine clay and/or pigment content (70% or more), containing 10-40 and/or pigment content (10%) or more), containing 10-40 pts. of a thermoplastic resin, dried to a moisture content of 5-10% and a second coating applied. The paper is again dried to 5-10%, brushed to bring out the gloss and finished by moulding the coated web against a polished metal surface with a resilient backing or between cooperating rollers, one highly polished and the other a resilient backing roll, at 5-35°F. above the second order transition temp. of the resin which may be an elastomer or a plastomer or a mixture of both.

Applications of Infrared Absorption Spectroscopy Investigations of Cotton and Modified Cottons (VI

XII-LEATHER; FURS; OTHER PROTEIN MATERIALS

Melamine and Resorcinol Resins for Finishing Leather K. Gutorski

Przegląd Skórzany (Biul. Lab. Kolor.), (7), 1-4 (1957):
Polish Tech. Abs., 29, (1), 147 (1958)
Finishes and coatings based on natural proteins still account, despite the increasingly frequent use of synthetic resins, for most of the output, since they make leather most delicate to the touch and do not erase the natural grain. Nevertheless, they still do not adequately protect the leather against the effects of water and other external factors. This shortcoming can be obviated by adding melamine or resorcinol resins. The various methods of obtaining particular resins, their advantages, and their shortcomings are discussed, and the application of some melamine and resorcinol resins for black and colours, finished on the basis of natural proteins, is described, and examples of finishes so obtained are given. The results prove that the use of melamine and resoreinol resins for opaque black and coloured finishes greatly enhances the resistance of leather to dry and wet friction, improves the lustre, and facilitates finishing operations. Use was made in protein finishes of two melamine resins produced by the Chemical Institute at Kedzierzyn—Melaform WM 100 and Melaform G. C.J.W.H.

Oxidation Dyeing of Furs F. Brugger and L.

Bór- és Cipótechnika, 7, (2), 39-41 (1957):

Hungarian Tech. Abs., 10, (1), 17-18 (1958) The influence of the conditions of oxidation dysing on the rate of dye development was studied in laboratory tests. The rate was, in general, increased by raising the tem-perature, but in the presence of chromium salts the maximum amount of oxidation products formed within the time usually allowed for fur dyeing at temperatures as low as 30°c. Therefore in the dyeing of furs without a mordant it is more advantageous to work at higher temperatures. To obtain the maximum yield of oxidation products, the necessary amount of oxidising agent is three equivalents for each mole of p-phenylenediamine, if chromium salts are present. In the absence of chromium salts even six equivalents of hydrogen peroxide will form only a fraction

E.V.T.

of the possible maximum yield of product. Potassium dichromate acts not only as an oxidising agent but also as a catalyst in dye development. Development of the colouring agent is catalysed equally by ter- and sexa-valent chromium. Consequently, a fur treated with dichromate mordant and exposed to the action of light, when chromium is reduced to the tervalent state, will be coloured in the same way as a fur not exposed to light. The degree of dispersion of the oxidation products is increased in the presence of ammonia.

Rabbit Fibres. XVIII—Frictional Properties of Angora Rabbit Fibres in Water

S. Ikeda, S. Okajima, and T. Inoue

J. Soc. Textile Cellulose Ind. Japan,

XIII-RUBBER; RESINS; etc.

14, 15-20 (Jan. 1958) Both frictional coefficients, the anti- and with-scale, increase when the fibres are degreased with CCl4. The effect of the thickness of fur on the frictional coefficient is negligible. C.J.W.H.

PATENTS

Lustring Furs

USP 2,799,599

BP 795,711

Askania Corpn. The fur is treated on the hair side with an organo-silicone polymer (preferably a silicone which is liquid at 20-25°c.) if desired together with oil and/or wax and then heated under pressure while simultaneously combing the hair.

Bating Hides or Skins after Treatment with a Protease

Röhm & Haas BP 795,850

Hides or skins which have been dehaired by use of a protease composition are enzymatically bated at pH 3-5. This results in leather having a firm grain. C.O.C.

Finishing Leather with Resin Dispersions

The leather is treated with an aqueous dispersion of a copolymer of an acrylic or methacrylic acid and an amide and then with formaldehyde. The dispersion can contain pigments, etc. The resultant finish has a natural appearance and is of excellent fastness to wet and dry rubbing. C.O.C.

XIII—RUBBER; RESINS; PLASTICS

Coated Fabrics-Manufacturing Processes Pigments used

H. Shepherd

J.S.D.C., 74, 449-457 (June 1958)

Methods and processes used in the coating of fabrics with linseed oil, india-rubber, nitrocellulose, and vinyl resin compositions are described. The preparation of the base cloths and the compounding of the coating media are described, together with spreading and calendering techniques of application. Embossing and decorative finishing are outlined, and recommendations on the choice of colouring matters are given.

PATENTS

Organotin Compounds as Stabilisers for Vinyl Halide Resins and the like

Metal & Thermit Corpn. Dibutyltin SS'-bis(2-ethylhexylmercaptoacetate) and dibutyltin SS'-bis(isooctylmercaptoacetate) are excellent stabilisers for vinyl halide resins. C.O.C.

Heat-stable Organic Cadmium Salts as Stabilisers for Vinyl Resin Compositions

National Lead Co. Use of basic Cd compounds with vinyl halide resins has the disadvantage that a deep yellow develops on prolonged heating. This is caused by formation of yellow cadmium oxide. Basic Cd salts of aliphatic monocarboxylic acids of > 5 C or an aromatic monocarboxylic acid when used with a small proportion on their weight of a salt of a glycol, aliphatic polyol, aliphatic polyhydroxy ether, an aminoglycol, a polyalkanolamine, an aliphatic hydroxy earboxylic acid, a polyhydroxy aromatic hydrocarbon or a polyhydroxy aromatic carboxylic acid all of 2-12 C, do not yellow upon heating for long periods and so can be used as stabilisers for vinyl halide resins without imparting any tendency for yellowing upon heating.

Printing Pastes, Sizes, Films, etc. (IX p. 671)

XIV-ANALYSIS; TESTING; APPARATUS Brazilin (C.I. Natural Red 24) as an Acid-Base Indicator

J. Bitskey and P. Móritz

Acta Chimica Academiae Scientiarum Hungaricae, 11, (3-4), 359-363 (1957):

Hungarian Tech. Abs., 10, (1), 3 (1958) A 1% alcoholic solution of Brazilin (from the South American brazilwood) can be successfully applied as an acid-base indicator for titrating strong acids and strong bases. The pH range is 5·85-7·73, the yellow colour changing to dark violet. Brazilin solution has the advantage that it can be stored for a long period of time without decomposition. Colour changes occur gradually at various pH values during titration, which indicates that the indicator might also be used for pH measurements. applicability of the indicator was proved by titrating several strong acids and a strong base with brazilin and, for reference, with Methyl Red (C.I. Acid Red 2). C.J.W.H.

Crystal Violet (C.I. Basic Violet 3) as a Reversible Indicator in Acetyl Chloride R. C. Paul, J. Singh, and S. S. Sandhu

Chem. and Ind., (21), 622-623 (24 May 1958) It is suggested that Crystal Violet can be used as an internal indicator for titration, in solutions in acetyl chloride, of Lewis acids such as TiCl₄ and SnCl₄ against bases such as quinoline, a-picoline, and dimethylaniline. The precipitate that is formed in all cases must be allowed to settle before the true colour of the solution can be

Polarographic Method for the Simultaneous Determination of Nitrite and Nitrate

P. Spanyár, E. Kevei, and M. Kiszel

Acta Chimica Academiae Scientiarum Hungaricae, 11, (3-4), 329-337 (1957): Aungarian Tech. Abs., 10, (1), 3 (1958)

To avoid the long time (about 50 min.) usually necessary for the decomposition of nitrous acid, it is liberated from nitrite in a medium containing hydrochloric acid and ethyl alcohol, and the ethyl nitrite formed is removed by a current of inert gas. The amount of nitrate may be determined in the known method after removal of nitrite. C.J.W.H.

Volumetric Determination of Dyes

E. Bene and K. Géczy

Acta Chimica Academiae Scientiarum Hungaricae, 11, (1-2), 49-56 (1957):

Hungarian Tech. Abs., 10, (1), 8 (1958) The method is based on the observation that certain dyes containing organic anions form compounds insoluble in water with some organic cation-active substances, but may be dissolved in some organic solvents. Chloroform was used as the organic solvent and cetylpyridinium bromide as the cation-active substance. The determination was carried out in a shaking cylinder. Titration with continued until the y colourless. The detercetylpyridinium bromide was aqueous layer became completely colourless. The determination of Chrysophenine G (C.I. Direct Yellow 12) in solutions of different concentrations, and examination of samples of Metanil Yellow (C.I. Acid Yellow 36) of Hungarian manufacture are presented as practical examples. The method gives very accurate results, the end-point of the titration being extremely sharp. C.J.W.H.

Plate Chromatography in Dye Analysis

G. Bussmann Schweiz. Apoth.-Ztg., 95, 915-9 (1957):

Chem. Abs., 52, 7705 (10 May 1958)
An account of the isolation and identification of watersoluble dyes by extracting them with quinoline followed by chromatography on Al₈O₈ plates. Comparison is made between plate and paper chromatography in the isolation of natural dyes. 22 references. C.O.C.

Direct Substitution of the Nitro Group in Aromatic Nitro Compounds by Chlorine using Carbon Tetrachloride

(IV p. 660) Effect of Velocity Gradient and Cellulose Concentration on Viscosities of Cellulose Solutions (XI p. 674)

Alkaline Solutions of the Iron-Tartaric Acid Complex (XI p. 674)

Molecular Size and Configuration of Cellulose Trinitrate in Solution (XI p. 675)

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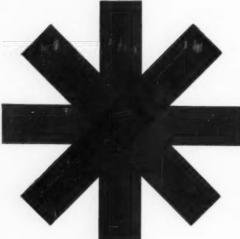
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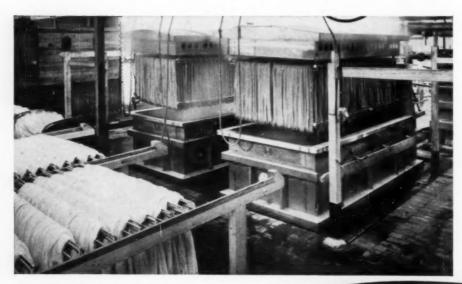
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FORTHCOMING MEETINGS OF THE SOCIETY - continued from page x

Friday, 21st November 1958

Bradford Junior Branch. The Dyeing of Blacks, Navy Blues and other Heavy Shades on Tricel by Azoic Methods. W. R. Beath, Esq., M.Sc., A.M.C.T. and J. S. Ward, Esq., B.Sc. (Courtaulds Ltd.). Institute of Technology, Bradford. 7.15 p.m.

HUDDERSFIELD SECTION. Annual Dinner. Princess Cafe,

Northumberland Street, Huddersfield.

MANCHESTER SECTION. Ladies Evening. College of Science and Technology, Manchester. Further details later.

Monday, 24th November 1958

HUDDERSFIELD SECTION. Invitation from The Halifax

Textile Society — Joint meeting at The Alexandra Hall, Halifax. 7.30 p.m.

Thursday, 27th November 1958
West Riding Section. Photoelectric Colorimeters— Their
Uses and Limitations in the Dychouse. J. V. Alderson,
Esq., B.Sc., A.R.C.S. (Imperial Chemical Industries
Ltd.). Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Tuesday, 2nd December 1958

Leeds Junior Branch. The Dyeing of Wool-Fibro Blends.

Miss E. Smith. Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, Leeds. 3.15 p.m.

Scottish Junior Branch. Some of the Principles and Practices of Textile Printing. A. Howarth, Esq., A.M.C.T., F.S.D.C., A.T.I. (The Seedhill Dyeing & Printing Co., Ltd.). Technical College, George Street, Paisley. 7.30 p.m.

Friday, 5th December 1958

BRADFORD JUNIOR BRANCH. Diamond Jubilee Dinner

LONDON SECTION. Dyeing of Furs. J. L. Stoves, Esq., M.Sc., Ph.D., F.R.I.C., F.C.S. (C. W. Martin & Sons Ltd.). The Royal Society, Burlington House, London W.1. 6 p.m.

Tuesday, 9th December 1958

Bradford Junior Branch. The Continuous Dyeing of Synthetic Fibre Blends. D. A. Garrett, Esq., B.A., A.Inst.P. and P. J. Dolby, Esq., B.Sc., F.C.S. Institute of Technology, Bradford. 7.15 p.m.

Northern Ireland Section. Dyeing Unions Containing Acrylic Fibres. B. Kramrisch, Esq., F.R.I C., F.T.I., F.S.D.C. (Ciba Clayton Ltd.). Grosvenor Rooms, Belfast. 7.30 p.m.

Scotting Section. Fast Shades on Wool-Cellulose Unions.

SCOTTISH SECTION. Fast Shades on Wool-Cellulose Unions. D. R. Lemin, Esq., B.Sc. (Imperial Chemical Industries Ltd.). St. Enoch Hotel, Glasgow. 7.15 p.m.

Wednesday, 10th December 1958 MIDLANDS SECTION. New Levelling Agents in Acid and Direct Dyebaths. G. H. Lister, Esq., B.Sc., Ph.D. King's Head Hotel, Loughborough. 7 p.m.

Thursday, 11th December 1958
WEST RIDING SECTION. The Development of a Dyestuff from its Synthesis to its Marketing. K. Roessler, Esq. (Badische Anilin & Soda-Fabrik). Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Tuesday, 16th December 1958

HUDDERSFIELD SECTION. The Development of a Dyestuff from its Synthesis to its Marketing. K. Roessler, Esq. (Badische Anilin & Soda-Fabrik A.G., Germany).

(Joint meeting with the Huddersfield Section of the Royal Institute of Chemistry.) Silvios Cafe, Huddersfield. 7.30 p.m.

Thursday, 18th December 1958

MANCHESTER JUNIOR BRANCH. Modern Package Dyeing.
A. Gibb, Esq. College of Science and Technology,
Manchester. 4.30 p.m.

Friday, 19th December 1958

MANCHESTER SECTION. Felisol— An International Guarantee of Colom Fastness. K. McLaren, Esq., B.Sc., F.R.I.C., F.S.D.C. (Imperial Chemical Industries Ltd.). The Textile Institute, 10 Blackfriars Street, Manchester. 7 p.m.

Tuesday, 6th January 1959

London Section. To Blend or not to Blend—That is the Question. Professor J. B. Speakman, D.Sc., F.R.I.C., F.T.I. (Leeds University). (Joint meeting with the London Section of the Textile Institute.) Chemical Society, Burlington House, London W.1. 6.30 p.m.

Thursday, 8th January 1959
WEST RIDING SECTION. New Levelling Agents in Acid and Direct Dyebaths. G. H. Lister, Esq., B.Sc., Ph.D. (Sandoz Products Ltd.). Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Friday, 9th January 1959

LONDON SECTION. Optical Brightening Agents in Action.
J. L. Ashworth, Esq., B.Sc., Ph.D., H. B. Mann, Esq.,
T. H. Morton, Esq., M.Sc., Ph.D., F.S.D.C., F.T.I.
(Courtaulds Ltd., Textile Research Laboratory, Bocking.) The Royal Society, Burlington House, London, W.1. 6 p.m.

Tuesday, 13th January 1959

Northern Ireland Section. Some Observations in the Uses of Synthetic Resin Products and Chemical Reactants to Cellulosic Materials. F. Sloan, Esq., M.Sc. (Kirkpatrick Bros. Ltd.). Grosvenor Rooms, Belfast. 7.30 p.m.

SCOTTISH SECTION. Dyeing Unions Containing Acrylic Fibres. B. Kramrisch, Esq., F.R.I.C., F.T.I., F.S.D.C. (Ciba Clayton Ltd.). St. Enoch Hotel, Glasgow.

7.15 p.m.

Thursday, 15th January 1959

MANCHESTER JUNIOR BRANCH. Management. Prof. R. W. Revans. College of Science and Technology, Manchester. 4.30 p.m.

Friday, 16th January 1959

MANCHESTER SECTION. The Degradation of Chemically Modified Celluloses by Alkali. Dr. W. M. Corbett (British Rayon Research Association). The Textile Institute, 10 Blackfriars Street, Manchester. 7 p.m.

Monday, 19th January 1959

HUDDERSFIELD SECTION. Title to be announced later.

Professor J. B. Speakman, D.Sc., F.R.I.C., F.T.I.,
F.S.D.C. (Joint meeting with the Huddersfield Textile Society.) Large Hall, Technical College, Huddersfield. 7.30 p.m.

Wednesday, 21st January 1959

MIDLANDS SECTION. The Solubility of Wool Dyes...

Assessment and Practical Significance. W. Beal, Esq.,
B.Sc. College of Technology, Leicester. 7 p.m.

Thursday, 22nd January 1959

West Riding Section. Some Aspects of Reactive Dyes. Dr. J. Wegmann. (Ciba Ltd., Basle). Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Friday, 23rd January 1959
LONDON SECTION. Annual Dinner and Dance. Waldorf
Hotel, London, W.C.2. 7 p.m. for 7.30 p.m.
WEST RIDING SECTION. Ladies' Evening. Victoria Hotel,

Bridge Street, Bradford.

Monday, 26th January 1959
BRADFORD JUNIOR BRANCH. Recent Developments in the use of Terylene Staple Fibre in the Woollen and Worsted Industry. P. W. Eggleston, Esq. (Imperial Chemical Industries Ltd.). Institute of Technology, Bradford 7.15 p.m.

Tuesday, 3rd February 1959
LEEDS JUNIOR BRANCH. Colour Matching. E. Bellhouse,
Esq., B.Sc. Lecture Theatre, Department of Colour
Chemistry and Dyeing, The University, Leeds. 3.15

Thursday, 5th February 1959

MIDLANDS SECTION. Developments in Modern Dry-cleaning Techniques. E. J. Davies, Esq., M.Sc. Gas Board Theatre, Nottingham. 7 p.m.

Friday, 6th February 1959
LONDON SECTION. Colour Photography. Dr. R. W. G. Hunt (Kodak Ltd.). The Royal Society, Burlington House, London W.1. 6 p.m. continued on page xl

FORTHCOMING MEETINGS OF THE SOCIETY—continued from page xxxix

Tuesday, 10th February 1959

Bradford Junior Branch. New Levelling Agents in Acid and Direct Dyebaths. G. H. Lister, Esq., B.Sc., Ph.D. (Sandoz Products Ltd.). Institute of Technology, Bradford. 7.15 p.m.

NORTHERN IRELAND SECTION. Emulsion Thickenings—Possibilities in Textile Printing. T. L. Dawson, Esq., B.Sc., Ph.D., A.R.I.C. (Imperial Chemical Industries Ltd.). Grosvenor Rooms, Belfast. 7.30 p.m.

SCOTTISH SECTION. The Use of Antistatic Agents in Textile Processing. A. E. Henshall, Esq., B.Sc. (Imperial Chemical Industries Ltd.). (Joint meeting with the Textile Institute.) St. Enoch Hotel, Glasgow. 7.15 p.m.

Thursday, 12th February 1959
WEST RIDING SECTION. New Ideas in the Application of Phthalocyanine Derivatives to Textiles. Dr. F. Gund. (Farbenfabriken Bayer). Griffin Hotel, Boar Lane,

Leeds. 7.30 p.m.

Tuesday, 17th February 1959

HUDDERSFIELD SECTION. The Use of Antistatic Agents in Textile Processing—Advantages and Disadvantages. A. E. Henshall, Esq., B.Sc. (Imperial Chemical Industries Ltd.) Silvios Cafe, Huddersfield. 7.30 p.m.

Thursday, 19th February 1959

MANCHESTER JUNIOR BRANCH. Chlorite Bleaching. W. J. Watts. Esq., R.Sc. College of Science and Technology,

MANCHESTER JUNIOR BRANCH. Chlorite Bleaching. W. J. Watts, Esq., B.Sc. College of Science and Technology, Manchester. 4.30 p.m. Friday, 20th February 1959

MANCHESTER SECTION. Emulsion Thickenings. Possibilities in Textile Printing. T. L. Dawson, Esq., B.Sc., Ph.D., A.R.I.C., F.C.S. (Imperial Chemical Industries Ltd.). The Textile Institute, 10 Blackfriars Street, Manchester. 7 n.m. 7 p.m.

Wednesday, 25th February 1959

Bradford Junior Branch. Vat Dyeing of Cotton Piece
Goods by Pre-pigmentation Routes. M. R. Fox, Esq.,
F.T.I., F.S.D.C. and J. F. Mawson, Esq., B.Sc.
(Imperial Chemical Industries Ltd.). Institute of Technology, Bradford. 7.15 p.m.

Technology, Bradford. 7.19 p.m.

Thursday, 28th February 1959

WEST RIDING SECTION. Fast Shades on Wool-Cellulose Unions. D. R. Lemin, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Victoria Hotel, Bridge Street, Bradford. 7.30 p.m.

Friday, 6th March 1959

LONDON, SECTION. The Me of Applicatic Appets in Taxille

LONDON SECTION. The Use of Antistatic Agents in Textile Processing. A. E. Henshall, Esq., B.Sc. (Imperial Chemical Industries Ltd.). The Royal Society, Burlington House, London W.I. 6 p.m.
MIDLANDS SECTION. Section Annual Dinner. George

Hotel, Nottingham.

Tuesday, 10th March 1959

LEEDS JUNIOR BRANCH. New Methods for the Assessment of Dyeing Properties and Usability of Dyes. G. H. Lister, Esq., B.Sc., Ph.D. Lecture Theatre, Department of Colour Chemistry and Dyeing, The University, Leeds. 3.15 p.m.

Leeds. 3.15 p.m.

Northern Ireland Section. Stenter Drying. T. A. Uthwatt, Esq., B.A., A.M.I.E.E., and J. S. Woollatt, Esq., B.Sc. (Mather & Platt Ltd.). Grosvenor Rooms, Belfast. 7.30 p.m.

Scottish Section. Annual General Meeting. 7 p.m. Followed by Modern Cloth Drying Principles and Machines. K. S. Laurie, Esq., A.M.I.Mech.E., A.M.I.E.E. (John Dalglish & Sons Ltd.). St. Enoch Hotel, Glasgow. 7-15 p.m.

Thursday, 12th March 1959

Bradford Junior Branch. Sizing Materials—Their Chemistry and Application. J. H. MacGregor, Esq., Ph.D., F.R.I.C., F.C.S. (Courtaulds Ltd.). Institute of Technology, Bradford. 7.15 p.m.

Late Correction

Wednesday, 29th October 1958
MIDLANDS SECTION. The lecture on Banlon Finishing is postponed. Instead, on this date Dr. G. H. Lister will give his lecture on New Levelling Agents which he was to have delivered on December 10th. It is hoped to give the Banlon lecture on December 10th.

Thursday, 12th March 1959 MIDLANDS SECTION. Dyeing and the Future. J. Boulton, Esq., M.Sc.Tech., F.R.I.C., F.T.I., F.S.D.C., President

Bag, M.Sc. 1ech., F.R.L.C., F. I.I., F.S.D.C., Frestdent of the Society. (Joint meeting with the Coventry Textile Society.) Courtauld's Acetate and Synthetic Fibres Laboratory, Lockhurst Lane, Coventry. 7 p.m. West Riding Section. The Sorption of Moisture by Fibre-forming Polymers. L. Valentine, Esq., B.Sc., Ph.D. (Tootal Broadhurst Lee Co. Ltd.). Victoria Hotel, Bridge Street, Bradford. 7,30 p.m. Saturday, 14th March 1959
Braddord Iunior Branch. Annual General Meeting.

BRADFORD JUNIOR BRANCH. Annual General Meeting. Institute of Technology, Bradford.

Tuesday, 17th March 1959

HUDDERSFIELD SECTION. Fast Shades on Wool-Cellulose

HUDDERSFIELD SECTION. Fast Shades on Wool-Cellulose Unions. Recent Advances in this Field. D. R. Lemin, Esq., B.Sc. (Imperial Chemical Industries Ltd.). Silvios Cafe, Huddersfield. 7.30 p.m.

Wednesday, 18th March 1959

MIDLANDS SECTION. Photoelectric Colorimeters...their uses and limitations in the Dychouse. J. V. Alderson, Esq., B.Sc. A.R.C.S. (Loist meetings with the British Association).

uses and limitations in the Dychouse. J. V. Alderson, Esq., B.Sc., A.R.C.S. (Joint meeting with the British Association of Chemists.) Midland Hotel, Derby. 7 p.m.

Thursday, 19th March 1959

MANCHESTER JUNIOR BRANCH. Films— Silk. Principles of Chromatography. How the World Looks to a Colour Defective. College of Science and Technology, Manchester. 4.30 p.m.

Friday, 20th March 1959

MANCHESTER SECTION. Symposium on Re-active Dyes. College of Science and Technology, Manchester. Further details later.

Further details later.

Further details later.

Thursday, 2nd April 1959

MIDLANDS SECTION. Colour Physics and Match Prediction.

(Lecturer to be announced later.) (Joint meeting with the Textile Institute.) Carpet Trades Ltd. Canteen, Kidderminster. 7 p.m.

Friday, 3rd April 1959
LONDON SECTION. Recent Developments in Dyeing.
T. Vickerstaff, Esq., M.Sc., Ph.D., F.S.D.C. (Imperial Chemical Industries Ltd.). George Hotel, Luton. 7 p.m.

Thursday, 9th April 1959 WEST RIDING SECTION. Annual General Meeting. Colour Film on How the World Looks to a Colour-defective. Victoria Hotel, Bridge Street, Bradford. 1

Wednesday, 15th April 1959 MIDLANDS SECTION. Annual General Meeting of the Section followed by a showing of New Scientific Films. King's Head Hotel, Loughborough. 7 p.m.

Thursday, 16th April 1959

MANCHESTER JUNIOR BRANCH. Three short papers by members of the section. College of Science and Technology, Manchester. 4.30 p.m.

Friday, 17th April 1959

MANCHESTER SECTION. Annual General Meeting. Stenter Drying. A. T. Uthwatt, Esq. (Mather & Platt Ltd., Research Dept.). The Textile Institute, Manchester.

Tuesday, 21st April 1959
HUDDERSFIELD SECTION. Annual General Meeting. Followed by a Colour Film on How the World Looks to a Colour-defective. Cmdr. Dean Farnworth (United States Naval Research). Silvios Cafe, Huddersfield. 7.30 p.m.

Friday, 24th April 1959 LONDON SECTION. Annual General Meeting. Commander Dean Farnworth (United States Navy) will show and discuss his film How the World Looks to a Colour-defective. Waldorf Hotel, London W.C.2. 6 p.m.

Late Addition

Saturday, 31st January 1959
Manchester Section. Annual Dinner and Dance,
Grand Hotel, Manchester. 6 p.m. for 6.30 p.m.

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8

APPOINTMENTS etc

Advertisements relating to Appointments Vacant, Appointments Wanted, and Miscellaneous Items are invited for insertion on this page. Advertisements of Appointments Wanted are gratis to members, but must not exceed twenty-four words.

All inquiries relating to Advertisements should be addressed to The General Secretary, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire.

Replies may be addressed Box —, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire, where all communications relating to these Advertisements are treated in strict confidence.

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AN EXPERIENCED DYER is required by large firm of Scottish dyers of fingering and hosiery yarns. Applicants must be first-class shade matchers, of strong personality, fully able to train and control labour, possess drive, initiative and a flexible outlook. This post is pensionable and offers wide scope. Apply in writing giving age, qualifications and posts held to Box 2682, Wm. Porteous & Co., dlasgow.

APPLICATIONS are invited for the position of Deputy Chief Chemist for Patons and Baldwins Limited, Darlington. Candidates must have qualifications of Honours Degree standard and should have experience in the application of dyestuffs and chemical finishes to natural and synthetic fibres.

Salary will be in accordance with qualifications and experience, and applications should be made to the Assistant Secretary, Patons and Baldwins Limited, Darlington.

ASSISTANT DYER

ASSISTANT DYER required for pressed felt industry in N.E. Lanes. Applicants should be 25 to 35 years of age with experience of dyelog wool and union piece goods. Sound practical performance is essential but technical qualifications will rate in salary assessment. Selected candidate would be expected to reside in the district. Sendful details to Box V154.

EXPERIENCED DYER required to take charge of sample dyeing of wool and synthetic fibres. Opportunity for man of initiative whose responsibilities will be varied by work in the Company's dyehouses. Pension scheme, canteen and five-day week. Apply by letter to—Assistant Secretary, Patons & Baldwins Limited, Darlington.

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Replies, giving details of qualifications and experience, to Box V155.

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